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FINAL REPORT

SEAL MATERIAL DEVELOPMENT PROGRAM
PHASE I

Prepared for

NASA Manned Spacecraft Center
Houston, Texas

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FOREWORD

This report presents the results of the first phase of a program to develop seal materials for oxygen-hydrogen propulsion systems. This program, "Seal Material Development," is planned to be a three-phase effort: Phase I, Material Development, Phase II, Seal Design Evaluation, and Phase III, Valve Test. The Phase I effort consisted primarily of identifying and characterizing advanced seal materials. The program was initiated in February 1970 and was conducted by the Science and Technology Division of TRW Systems, Redondo Beach, California, for the NASA Manned Spacecraft Center, Houston, Texas, under contract NAS 9-10481. The program was conducted under the technical direction of Mr. M. C. Buchanan of the NASA Manned Spacecraft Center. Mr. F. E. Compitello is the NASA Headquarters Project Manager. The TRW Systems Program Manager is Mr. R. W. Wright. Performance of the program was actually conducted at three locations: TRW Systems, Redondo Beach; TRW Capistrano Test Site, Capistrano, California; and Wyle Laboratories, Norco, California.

Substantial support and assistance was provided by the Chemical Engineering Department, Dr. E. Burns, Manager. All material or compounds used in this program were either compounded or obtained by the Chemical Engineering Department. All materials with an AF-E designation were used with the approval and assistance of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Dayton, Ohio.

Acknowledgement is also made of other major contributors to this program. Mr. J. W. Martin, material compounding and evaluation, Mr. J. R. Denson for LN_2 and LO_2 capability tests, and Mr. R. G. Gilroy technical assistance and final report preparation.

ABSTRACT

The Seal Material Development Program is planned as a three-phase program. This report covers Phase I which was conducted to identify and characterize materials which would advance the state of the art for oxygen-hydrogen propulsion system seals. The temperature range to be covered was from -423°F (37°R) to +200°F (660°R). A total of nineteen materials were evaluated, with fourteen being tested at least once during the program. Included in the materials tested were Teflon (as the control material), ethylene propylene terpolymer compounds, two silicone compounds, HYSTL (a TRW polyurethane resin), an experimental fluorinated elastomer (AF-E-124D), and Viton-A. Although the goal of the program was to identify a single material for use in both oxygen and hydrogen over the entire temperature range, a number of materials were characterized for use in either oxygen or hydrogen.

The experimental fluoroelastomer AF-E-124D generally indicated superior performance to other materials for use in both oxygen and hydrogen systems. The Teflon TFE performed satisfactorily in both propellants. Viton A and HYSTL exhibited excellent seal characteristics for use in liquid hydrogen systems. Tests conducted included liquid hydrogen seal tests, ABMA LO_2 impact sensitivity tests, and cleaning and flushing fluid and elevated temperature exposure tests.

Further characterization of AF-E-124D as well as other promising materials is recommended to establish design parameters for these materials. In addition, fabrication and test of materials in various seal configurations are the next steps in developing promising materials as advanced oxygen-hydrogen propulsion system seals.

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1. SUMMARY

The Seal Material Development Program, Phase I, was conducted to identify materials which would significantly advance the state of the art in seals for oxygen-hydrogen propulsion systems. This requires materials compatible with hydrogen and oxygen at temperatures from -423°F (37°R) to +200°F (660°R). The mechanical properties must be such that the parameters important to seal materials are retained over this temperature range. Consequently, the superior ambient and elevated temperature sealing capability of elastomers strongly indicated that this program be oriented toward identifying elastomers with acceptable low temperature sealing and mechanical properties.

Although the ultimate goal of this program was to identify a single material capable of meeting the specification requirements, it was acknowledged that this material would not necessarily be superior to all others in all respects. Because of this, most of the selected materials were retained for evaluation throughout the program to provide a rating for combined LO_2 and LH_2 service or for either propellant. The relative ranking of the materials tested in this program and also of other typical cryogenic high temperature compounds is presented in Table 1-1.

As a result of the program evaluations, a single material which exhibited the desired properties did evolve, and, in general, was superior to the control material Teflon with both LH_2 and LO_2. This material, an experimental fluoroelastomer designated as AF-E-124D, has proven to be very promising in all the tests conducted during this program. The tests performed included LH_2 valve seal tests, ABMA LO_2 impact tests, solvent and flushing fluid compatibility tests and elevated temperature tests (to +400°F).

The AF-E-124D material used in this program is a pure material with no fillers or other additives. Continuing development by TRW on other programs includes the compounding of this material with other additives to establish which properties can be further improved. The material tested may be considered the "base" polymer from which a series of compounds will be developed. These new compounds should be evaluated

Table 1-1. Summary of Seal Material Performance

Rating			Material	Sealing Capability Ratio of Load Required to Seal at Cryogenic Temperature to Ambient Temperature		LO_2 Impact Resistance Maximum Impact with no Reaction ft-lbs	Resistance to Permanent Seat Deformation (3)	High Temperature Resistance, Average Percent Change in Tensile Strength	
H_2	O_2	O_2/H_2		LH_2	LN_2		+60 to -423°F	+400°F	+250°F
2	1	1	AF-E-124D	6.0	6.5	72	Excellent	-20	-27
4	2	2	Teflon TFE	10.0	3.7	72	Fair	0	0
3	6	6	255-2 (Viton A)	5.0	4.5	40	Good	0	-12
8	7	7	310-1 (Phenyl Silicone)	4.7	3.4	<20	Good	-26	-14
1	***	***	HYSTL	3.7	2.9	<10	Excellent	0	0
**	***	***	316-1 (Fluoro-silicone)	**	12.5	<10	Poor	-50	-28
12	***	***	AF-E-71-2	11.6	6.0	<10	Fair	-93	-54
11	***	***	263-2 (EPT-HYDRIN)	6.3	(1)	<10	Fair	*	0
9	***	***	Mylar (2)						
5	3	3	Kel-F (2)						
16	***	***	Nylon (2)						
7	5	5	Kynar (2)						
6	4	4	Vespel (2)						

*Material Decomposed.

**Material failed under load.

***Not rated due to LO_2 impact sensitivity

(1) Not tested.

(2) Not tested during this program

(3) Definition of terms:
Excellent - No evidence of permanent set
Good - Slight seat imprint
Fair - Medium to deep seat imprint
Poor - Seat cracked

with respect to seal capability since significant improvements would be anticipated in tensile strength, compression set, low temperature toughness, and elevated temperature resistance.

Since AF-E-124D was only available late in the program, and in limited quantities, a complete characterization was not possible. Sufficient representative tests were completed to provide an indication of the overall superiority of this material. The future investigation recommended for one of the items included in the Phase II effort is the full characterization of this material both at cryogenic and elevated temperatures. Also recommended is the fabrication of poppet seats and static seals for valve propellant tests with Viton-A, and HYSTL using Teflon as the control.

2. INTRODUCTION

The objective of this program was to advance cryogenic sealing technology through the use of improved seal materials. Although the initial effort included a wider range of propellants, the program was redirected soon after initiation, focusing on hydrogen and oxygen. The goal of the program was to find a single polymer capable of sealing both propellants. The desired material had to function as an effective seal in a variety of static and dynamic seal applications over a temperature range of -423°F to +200°F. Although the maximum temperature specified in the contract was +200°F, all materials were evaluated up to 400°F to emphasize the differences in their elevated temperature properties for comparison purposes.

Teflon was selected as the standard against which to compare other candidate polymers, since it is presently in use and is well characterized. Emphasis was placed on evaluating elastomers because of the inherently better sealing characteristics as compared with plastics (i.e., compliance and resilience).

This report covers Phase I of a seal material development program. Materials selected, and an experimental evaluation effort to screen a variety of polymers for potential service as cryogenic seals are described. The ultimate application of a successful seal material includes seats, o-rings, lip seals, slipper seals and packings used in check valves, static flanges, ball valves, regulators and solenoid valves.

Based on the success of the Phase I, Material Development, subsequent phases planned are Phase II, Seal Design Evaluation, and Phase III, Valve Test.

A number of referencs which were used in the preparation of this report are listed at the end of the report.

3. PROGRAM DESCRIPTION

3.1 PROGRAM REQUIREMENTS AND GOALS

The basic objective of this program was to identify and evaluate conventional and advanced seal materials for use in cryogenic propulsion system applications. Table 3-1 summarizes the functional parameters, seal applications and other constraints which were included in the program investigations and are pertinent to propulsion system requirements.

Table 3-1. Seal Material Requirements

Propellants	Liquid Oxygen and Liquid Hydrogen
Temperature Range	-423°F to +200°F (extended to +400°F)
Seal Types	Poppet Seats, O-Rings, Lip Seals, Slipper Seals
Leakage Rate (smallest diameter seals)	10 scc/hr or less
Cleaning Fluids	Distilled water, Freon TF, Isopropyl Alcohol, Trichloroethylene

There are a number of existing techniques and methods used to obtain a seal at cryogenic temperature. However, each state-of-the-art technique has a deficiency which limits its application in some way. The more obvious method of sealing at cryogenic temperatures is the use of a metal to metal seal. This generally eliminates the concern over seal material degradation or properties of non-metallics. This technique is commonly used with success at both ambient and cryogenic temperatures. Metal to metal seals, however, have some deficiencies. The metal to metal seal is contamination and wear sensitive, and the larger the seating area, the greater the possibility of contaminant lodging between the seat and poppet of a valve, resulting in leakage. Other problems with this technique include: high seat load requirements, seat abrasion resulting in leakage, and unequal contraction/expansion of seat causing distortion and subsequent leakage. The use of plastics such as Teflon and Kel-F is also common, overcoming some of the problems of the hard seat

approach, and are more tolerant to imperfections in the design or system generally. In a cryogenic propulsion system, the valve seat or static seals must be capable of withstanding elevated temperatures as well as low temperatures. With plastics such as Teflon, an unacceptable degree of cold flow occurs at elevated temperatures which drastically reduces the seal life.

The ideal cryogenic seal material is one which can be used in both liquid oxygen (LO_2) and liquid hydrogen (LH_2), as well as the various cleaning and test media normally employed. A necessary requirement of a cryogenic propulsion system seal material is that it be capable of operating in the propellant, and that it can be cleaned and flushed with solvents and operate at cryogenic temperatures as well as the elevated temperatures resulting from heat soakback. The objective of this program was to obtain the "ideal" material; or, at least, provide a separate material for each of the propellants (LO_2 and LH_2).

3.2 PROGRAM APPROACH

Realizing the problems inherent with existing cryogenic seal technique (all metal and plastic), the approach taken by this program was basically the investigation of elastomeric materials, and the identification/development of promising elastomers. This plan was adopted because of the generally superior sealing characteristics of elastomers at ambient and elevated temperatures, which is where the bulk of seal degradation occurs. It was also used because of preliminary investigations conducted at TRW, using elastomers compounded with a resin, HYSTL, which exhibited excellent ambient and low temperature properties. Since Teflon is generally recognized as the state-of-the-art cryogenic seal material, it was used as the control material from which other data is referenced.

Essentially, the program approach was to identify or compound an elastomer which, while retaining the desirable elastomeric characteristics at ambient and elevated temperature, also exhibited a degree of resiliency at cryogenic temperatures.

The program consisted of three major tasks. These were:

Task I	Evaluation of materials, selection of test materials and formulation of test plans
Task II	Test Program
Task III	Data review, correlation and application to seal design; final report.

During Task I, Material Selection, existing data on candiate materials were obtained and reviewed and a literature search was conducted, reviewing both published and unpublished documents and data. As a result of this review and evaluation of data, materials were selected for testing in the Task II effort. At this point, a more definitive test program was established. To make maximum use of the resources on the program it was decided to run extensive tests on a few promising materials rather than to do limited screening of a great many materials. Thus, the basic goal in selecting candidate materials and tests to be conducted was to select either new compounds or those which had limited or no low temperature test data, and yet theoretically provided promise as a cryogenic and elevated temperature seal material in both LO_2 and LH_2. For this reason, some common state-of-the-art cryogenic seal materials were not tested because sufficient data presently exists. However, these materials were considered in the overall evaluation and in selecting materials for continuing work in Phase II. During Task II, Materials Testing, the evaluation and screening of additional candidate materials continued, with some previously selected materials being deleted and other more promising materials added. A list of all the materials tested is shown in Table 3-2 with a brief statement of relative capability or reason for deletion.

The tests conducted during this task were:

- Liquid Nitrogen (LN_2) Seal Load Retention Test
- LO_2 Compatibility Tests
- LN_2 Valve Seal Tests
- ABMA LO_2 Impact Tests
- LH_2 Valve Seal Tests
- Cleaning Fluid Exposure Tests
- Elevated Temperature Tests

Table 3-2. Program Summary Chart

MATERIAL SELECTIONS		LN_2 COMPRES. LOAD TESTS	LO_2 COMPAT. SUBMERS. TESTS	LN_2 (4) TEMP. FLEX. TESTS	LN_2 TEMP. VALVE TESTS	SOLVENT EXPOSURE TESTS	ABMA LO_2 IMPACT TESTS	HIGH TEMP EXPOSURE TESTS	LH_2 TEMP. VALVE TESTS
PROPOSAL SELECTIONS	TEFLON	GOOD	GOOD		GOOD	EXCEL	EXCEL	EXCEL	FAIR
	EPR	(2)							
	POLYURETHANE	N/A	(3)						
	NITROSO	N/A	N/A		FAILED				
	HYSTL	N/A	GOOD		EXCEL	GOOD (5)	POOR	GOOD	EXCEL
TASK I SELECTIONS	KEL-F (1)								
	MYLAR (1)								
	NYLON (1)								
	POLYIMIDE (1)								
	BUNA-N	POOR	GOOD		FAILED				
	POLYBUTADIENE	POOR	GOOD		FAILED				
	POLYETHYLENE	N/A	N/A						
	AF-E-71-2 (EPT)	GOOD	GOOD	-40° F	GOOD	FAIR	POOR	POOR	FAIR
	VITON-A	GOOD	GOOD		GOOD	EXCEL	GOOD	GOOD	GOOD
SUBSEQUENT ADDITIONS	FLUOROSILICONE	N/A	GOOD		POOR	FAIR	POOR	FAIR	FAILED
	PHENYL SILICONE	GOOD	GOOD	-170° F	FAIR	FAIR	FAIR	GOOD	FAIR
	EPT-HYDRIN	GOOD	GOOD		GOOD	GOOD	POOR	POOR	FAIR
	AF-E-124D	N/A	→	→	EXCEL	FAIR	EXCEL	GOOD	GOOD

NOTES

(1) NOT INCLUDED IN TEST PROGRAM DUE TO AMPLE AVAILABLE DATA AND THE DECISION TO CONFINE TEST PROGRAM TO ELASTOMERS (SEE REFERENCES)

(2) REPLACED WITH AF-E-71-2 AND EPT-HYDRIN

(3) ELIMINATED FROM PROGRAM DUE TO HIGH COST AND POOR AVAILABILITY

(4) ONLY LIMITED TESTING CONDUCTED DUE TO INCONSISTENCIES

(5) DECOMPOSED IN TRI-CHLOROETHYLENE

N/A - NOT AVAILABLE

Each of these tests are discussed in detail with a summary of test results in Section 5.

In all the test series, the emphasis was placed on obtaining data relative to the bulk material capabilities in the various test media or environments. This provides a very conservative approach to material selection in that results obtained are the results provided by the material itself in bulk form. In this way, a specific seal or material configuration is not tested, which could bias results dependent on the seal design. From this point of view, the minimum capabilities of the material are reflected, which can be improved when used as a seal, by designing specifically to emphasize the strong characteristics and minimize the weak areas.

In Task III, the data accumulated as a result of the literature search and testing was evaluated to establish those materials most likely to satisfy the program goals.

A summary of data is provided in Appendices A, B, and C. Of necessity, a great deal of the evaluation must be subjective since materials properties cannot always be directly compared, although to a maximum extent, conclusions are supported by the quantitative data. One problem area is the comparison of test data generated in this program with that from the literature. The tests conducted under this program were oriented toward identifying those characteristics most applicable to seal design while data from the literature is not necessarily so oriented.

4. MATERIALS SELECTION

4.1 SEALING CRITERIA — INFLUENCE ON DESIRED MATERIALS CHARACTERISTICS

The fundamental function of a seal is to block all macroscopic leak paths by a continuous mating of the sealing surfaces. Continuous mating means achieving unbroken contact along the entire sealing surface and maintaining this contact after the initial seal has been made, i.e., the ability to retain load at the sealing interface despite relative movement of the joint surfaces caused by changes in the operating environment. Environmental influences that can bring about these changes include pressure variation, temperature changes and externally applied loads. The primary characteristic of a seal material which provides continuous sealing load is its resilience or elasticity. Although there are a number of ways to design elasticity into a seal, the most direct way is for the seal material itself to be elastic.

Intimate mating between two surfaces can be achieved by perfect matching through techniques such as carefully finishing the surfaces so that they mate geometrically. This, however, is not easy to accomplish. An easier means of achieving mating of surfaces is for one surface to be relatively soft and compliant so that it conforms to the irregularities of the mating surface. With regard to both desirable characteristics, resilience and compliance, elastomers as a class of materials uniquely fulfill these requirements. Although certain plastics (including Teflon) are compliant, these materials often lack dimensional stability or elastic memory resulting in a tendency for the material to relax or lose its sealing load with time. This characteristic is the commonly referred to "cold flow" which must be carefully considered by the designer of the seals when utilizing a material such as Teflon.

Two factors have limited the use of elastomers for LO_2 and LH_2 sealing applications. At low temperatures elastomers become hard and brittle, and most elastomers cannot withstand the strong oxidizing environment of liquid oxygen, particularly under impact conditions. Because little work has been done in the utilization of elastomers at low temperatures, the primary effort of this program was directed toward determing

if a material with elastomeric properties could be found for application with liquid hydrogen and liquid oxygen. It is also important to note that even though elastomers become hard at low temperatures, they have the distinct advantage over plastics of improved sealing at normal ambient temperatures and above. For cryogenic seal applications, Teflon was selected as the baseline material against which to compare other candidate materials. Other state of the art materials utilized for low temperature sealing are Kel-F (plastic), Mylar and Nylon. Limited use has also been made of Kynar and Vespel. Teflon is the most compliant (most like an elastomer with respect to ease of sealing). The other plastics are significantly harder than Teflon and with the exception of Kel-F, are impact sensitive in liquid oxygen. Kel-F is probably second to Teflon in use as a low temperature seal material; it has a higher strength, is more resistant to cold flow, but is brittler and generally requires higher sealing loads.

However, as Kel-F has been well characterized and its usefulness as a seal material in comparison with Teflon is limited, the material was not included in the present test program. Likewise due to the emphasis on compliant materials, Mylar, Nylon, Kynar and Vespal were not tested.

4.2 GLASS TRANSITION TEMPERATURE

As the temperature of a polymer is reduced, a point is reached where the material undergoes a transition to become hard and rigid. This point, known as the glass transition temperature (Tg), is above -200°F for all known polymers. A more significant factor regarding the Tg is that, once below this temperature, the general physical properties of a material do not change as rapidly with decreasing temperature. Use was made of this characteristic by conducting the majority of the test program with LN_2, a much safer and less expensive cryogen than LO_2 or LH_2. The expectation was that the results would not differ significantly at the lower LH_2 temperatures. This assumption was subsequently proven to be true. Some polymers retain toughness below their glass transition temperature as contrasted with others which become brittle. The ability of a material to retain toughness at cryogenic temperatures may be its composition rather than its low temperature resistance characteristic, Tg. Specifically, the neat polymers, polymers having no fillers or additives (Teflon, HYSTL, AF-E-124D), showed good sealing ability and resistance to brittle failure under impact.

The one elastomer included in the program which is a neat polymer and showed promise as a cryogenic seal was AF-E-124D.

4.3 MATERIAL SELECTION CRITERIA

The general classes of elastomers considered for evaluation included the fluorocarbon elastomers, silicones and hydrocarbons. Two primary factors were considered initially in the selection of elastomeric base polymers. The first was low temperature flexibility and the second was high temperature oxidation resistance. In general, hydrocarbons and silicones best meet the first criterion. Similarly, fluorocarbons and silicones best meet the second criterion. Although silicones have poorer engineering properties, these properties could be improved somewhat through compounding. Thus, the elastomers chosen for primary evaluation included representative hydrocarbons, fluorocarbons and silicones. The two exceptions to the elastomer criteria for materials selection (other than the Teflon control) were HYSTL and polyethylene. Table 4-1 describes all the materials included in the test program. The rationale for considering specific materials is presented below.

4.3.1 Fluorocarbon Elastomers

Several specific compounds in this category of elastomers are of primary interest because of the relative chemical inertness of the materials in the presence of a strong oxidizer. In fact, the fluorocarbons are the only general class of polymers which are known to be insensitive to impact in the presence of oxygen. Three fluorocarbon elastomers were selected for evaluation: (1) Viton A is a well known elastomer used extensively for vacuum sealing applications; (2) Carboxynitroso rubber is a relatively new perfluorinated elastomer having both an excellent resistance to strong oxidizers at high temperature and an unusually low glass transition temperature (for a fluorocarbon); and (3) AF-E-124D is an experimental fluorinated elastomer. Kel-F elastomer, which is very similar to Viton, was eliminated primarily because it has an extremely high strain relaxation rate. That is, it like Teflon, suffers from cold flow characteristics. Kel-F elastomer also has a relatively high glass transition temperature.

Table 4-1. Materials Selected for Evaluation

	Material	TRW Compound Designation	Base Polymer Identification	Description
1.	Teflon	Teflon-TFE	TFE DuPont	Tetrafluoroethylene M1-P-19468
2.	EPT/HYSTL	AF-E-71-2	Nordel 1040, DuPont[a]	Blend of ethylene/propylene terpolymer and HYSTL
3.	Viton-A	255-2	Viton-A, DuPont	Copolymer of Hexafluoropropylene and vinylidene fluoride
4.	EPT/HYDRIN	263-2	Nordel 1040, DuPont HYDRIN 200, B. F. Goodrich	Blend of ethylene propylene terpolymer and epichlorohydrin
5.	Nitrile	215-2	Chemigum N-9, Goodyear	Nitrile rubber, copolymer of butadiene and acrylonitrile
6.	Polybutadiene	60-7	Cis-4, Phillips Petroleum	Cis 1, 4-polybutadiene
7.	Silicone	310-1	Dow Corning 440	Methyl/vinyl/phenyl siloxane
8.	Fluorosilicone	316-1	Dow Corning LS-63	Hexafluoropropyl/methyl/vinyl siloxane
9.	HYSTL	HYSTL		Polybutadiene Urethane
10.	Experimental Fluoroelastomer	AF-E-124D	(b)	(b)
11.	Polyethylene			
12.	Carboxynitroso rubber	299-1	Pennisular Chemresearch Lot CNR-4	Carboxynitroso rubber cured by HYSTL Resin

[a] EPT is the continuous phase, HYSTL is the dispersed phase.

[b] Believed to be a Terpolymer of perfluorovinyl ether/TFE and an undisclosed cure group.

4.3.2 Silicones

Silicones are unique elastomers in that they have low glass transition temperatures and are generally useful to considerably lower temperatures than other elastomeric materials. They are also resistant to high temperature oxidation.

4.3.3 Hydrocarbons

Specific hydrocarbon elastomers included for evaluation were nitrile rubber, polybutadiene and ethylene propylene terpolymers. Nitrile rubbers, although having a relatively high glass transition temperature, are known to be inherently tough materials even below their glass transition temperatures. Polybutadiene was selected for evaluation based on its unusually low glass transition temperature (-145°F).

Ethylene propylene terpolymer (EPT), although known to be degraded by oxygen at elevated temperatures, was mixed with a halogenated carbon polymer with the possibility that its resistance to oxidation could be improved. Polyethylene, which is one of the constituents of EPT, has an unusually low glass transition temperature (-190°F). The copolymers of ethylene theoretically can approach the glass transition temperature of polyethylene provided they are of an appropriate ethylene/propylene ratio and have limited chain branching in their microstructure. Ethylene/propylene terpolymer (Nordel 1040, DuPont EPT) approaches these requirements. The two EPT compounds selected were EPT/HYDRIN and AF-E-71-2. HYDRIN is a halogenated polymer known to have fire extinguishing properties and considered to be oxygen compatible. HYSTL has shown excellent resistance to strong oxidizers and rocket fuels. HYSTL was developed by TRW under contract to NASA (NAS3-4188). Its unusual chemistry makes it possible to effectively blend HYSTL with other polymers. AF-E-71-2 is such a blend, a combination of HYSTL and ethylene propylene rubber. The blend is elastomeric in nature even though neat HYSTL is a hard plastic. HYSTL is a high-carbon resin, poly (1, 2 polybutadiene) tolyl urethane.

4.3.4 Polyethylene

Polyethylene was included because of its unusually low glass transition temperature (-190°F).

4.3.5 HYSTL

HYSTL was included because it can be co-vulcanized to provide an elastomer (EPT/HYSTL) and has shown excellent resistance to strong oxidizers. Also, HYSTL, like Teflon and polyethylene is a neat material, having no fillers, plasticizers, or other additives. The presence of these ingredients is postulated to have an adverse effect on the low temperature properties of a polymeric material.

4.4 MATERIALS PROPERTIES

Table 4-2 presents a summary of the properties of the materials, considered for inclusion in the experimental program. Mylar, Kel-F, Nylon, Kynar and Vespel were not included in the experimental program for the reasons cited earlier. Of the materials selected for testing with the exception of Teflon and polyethylene, all the polyners are thermosets. These materials have high crosslinked, three-dimensional, chemically bonded molecular networks. These compounds in contrast to thermoplastics (such as Teflon) resist the tendency to cold flow under stress because intermolecular slippage is counteracted by chemically bonded crosslinks.

4.5 COMPOSITION OF SELECTED COMPOUNDS

Table 4-3 gives the formulation of compounds prepared by TRW for evaluation as cryogenic seals. It is important to realize that both the mechanical and chemical properties of an elastomeric base polymer are changed significantly by compounding, and pure elastomeric base polymers are almost never used to meet engineering requirements. In general, the compounds shown in Table 4-3 are characterized by low volumes of fillers (better compression set), silicon dioxide filler (resistant to oxidation), chemically stable crosslink networks (initiated by peroxide rather than sulfur, except for the nitrile) and no plasticizers which could be extracted by the propellants and result in a change in sealing properties.

Table 4-2. Summary of Properties of Material Evaluated

Material	Compound Number	Tensile Strength (psi)	Hardness (Shore A)	Modulus (psi)	Elongation at Break (%)	Tensile[c] Set (%)	Approximate Glass Transition Temperature (°F)
Teflon	TFE	3,000-4,000	Rockwell 58R	580,000	200	---	+77
EPT/HYSTL	AF-E-71-2	1,800	90	1,425[b]	110	2	-50
Viton-A	255-2	2,400	80	350[b]	350	10	-50
EPT/HYDRIN	263-2	1,200	80	---	120	10	a)
Nitrile	215-2	2,500	70	700[b]	325	6	-45
Polybutadiene	60-7	1,400	75	250[b]	440	10	-145
Phenyl Silicone	310-1	900	70	---	140	---	-125
Fluorosilicone	316-1	840	65	---	165	---	-78
HYSTL		5,000	30-40 Barcol	---	5	---	+450
Exp. Fluoro-elastomer	AF-E-124D	2,600	78	835[b]	150	2	+20
Nitroso	299-1	725	80	350[b]	160	4	-65
Polyethylene, high density		2,500	Rockwell 50R	60,000-150,000	50	---	-190
Mylar		23,000	---	550,000	100	---	+156
Kel-F		4,500-6,000	Rockwell 110-115R	150,000-300,000	125-175	---	+113
Nylon		7,000-12,000	Rockwell 111-118R	260,000-400,000	50-180	---	+134
Kynar		7,000	Shore D70	120,000	100-300	---	-49
Vespel		10,500	Shore D 70-90	430,000	6-8	---	+470

a) Will be determined by the elastomer specie which is the continuous phase.
b) at 100% elongation
c) Per ASTM D412; 20 in/min strain rate; measured 30 min after pull test

Table 4-3. Compositions of Elastomer Compounds Selected for Evaluation

Compound - 60-7	AF-E-71-2	215-2	255-2	263-2	310-1	316-1
Type - Polybutadiene	EPT	Nitrile	Viton A	EPT/Hydrin	Phenyl Silicone	Fluorosilicone
Unsat. hydrocarbon	Sat. hydrocarbon	Hydrocarbon copolymer	Fluorocarbon	Hydrocarbon blend	Polysiloxane	Polysiloxane
cis 1, 4-Polybutadiene 100	Air Force proprietary	Chemigum N-9 100	Viton A 100	Nordel 1040 70	D.C. 440 100	LS 63 100
Cab-O-Sil M-5 40	Developed by TRW Systems	Cab-O-Sil M-5 40	Ca O 15	Hydrin 200 30	B-3000 Resin 15	Di-Cup R 0.75
Di-Cup R 2.5		Zinc Oxide 2	SRF 20	Cab-O-Sil EH-5 30	Teflon T-7 10	
		Santocure NS 1	Diak #3 1.5	C-100 Resin 14	Cab-O-Sil M-5 20	
		Sulfur 1.5		Di Cup R 6	Di-Cup R 6	
				Zinc Oxide 2		
				NBC 1		
Cure - 10'/320°F		15'/325°F	30'/325°F	NA 22 1.5	30'/330°F	30'/320°F
Post Cure - 2 hr/225°F		2 hr/225°F	24 hr/400°F	30'/350°F	24 hr/400°F	6 hr/400°F
				2 hr/225°F		

NOTE: Compound 263-2 is a blend of two compounded base elastomers each with their respective curatives included.

5. TEST DESCRIPTIONS AND RESULTS

5.1 LN_2 TESTS

The LN_2 test program was designed to serve a dual purpose as the initial phase of the cryogenic evaluation portion of the program. The test series was intended to screen out materials not suitable for cryogenic service, and to determine the general cryogenic characteristics of the acceptable materials at LO_2 temperatures. For this purpose, liquid nitrogen was selected because its boiling temperature of -320°F is close to that of LO_2, -297°F; it is inexpensive, easy to handle and readily available. Also, a review of previous research on polymers at cryogenic temperatures indicated that only minimal changes in physical characteristics would be expected at progressively lower temperatures below the glass transition temperature. Consequently, the use of LN_2, at -320°F, was considered warranted for screening tests to evaluate anticipated properties at LH_2 temperatures of -423°F. This theory was proven to be generally correct since the relative ranking of materials generated from the LN_2 test series did not materially change when tested with LH_2.

The test series was specifically designed to evaluate low temperature sealing characteristics, as contrasted with the usual approach of determining physical properties which have been of only rudimentary help to the valve designer. Good cryogenic sealing characteristics are primarily dependent on the ability of the material to maintain an initially applied preload throughout the operational temperature range. The LN_2 test series was programmed to obtain quantitative data from which the relative load retention characteristic of the selected materials could be compared. The test program was divided into two distinct segments; compression load tests for the determination of the low temperature load retention properties; and valve seal tests designed to measure directly the load required to effect a seal. Load retention is an important criteria for static seals, whereas seal loading is a necessary parameter for poppet seal designs, two of the more common types of seal applications.

5.1.1 Compression Load Tests

The test sequence adopted to determine the ability of a material to retain an initially applied load together with an idealized load-deflection curve, is presented in Figure 5-1.

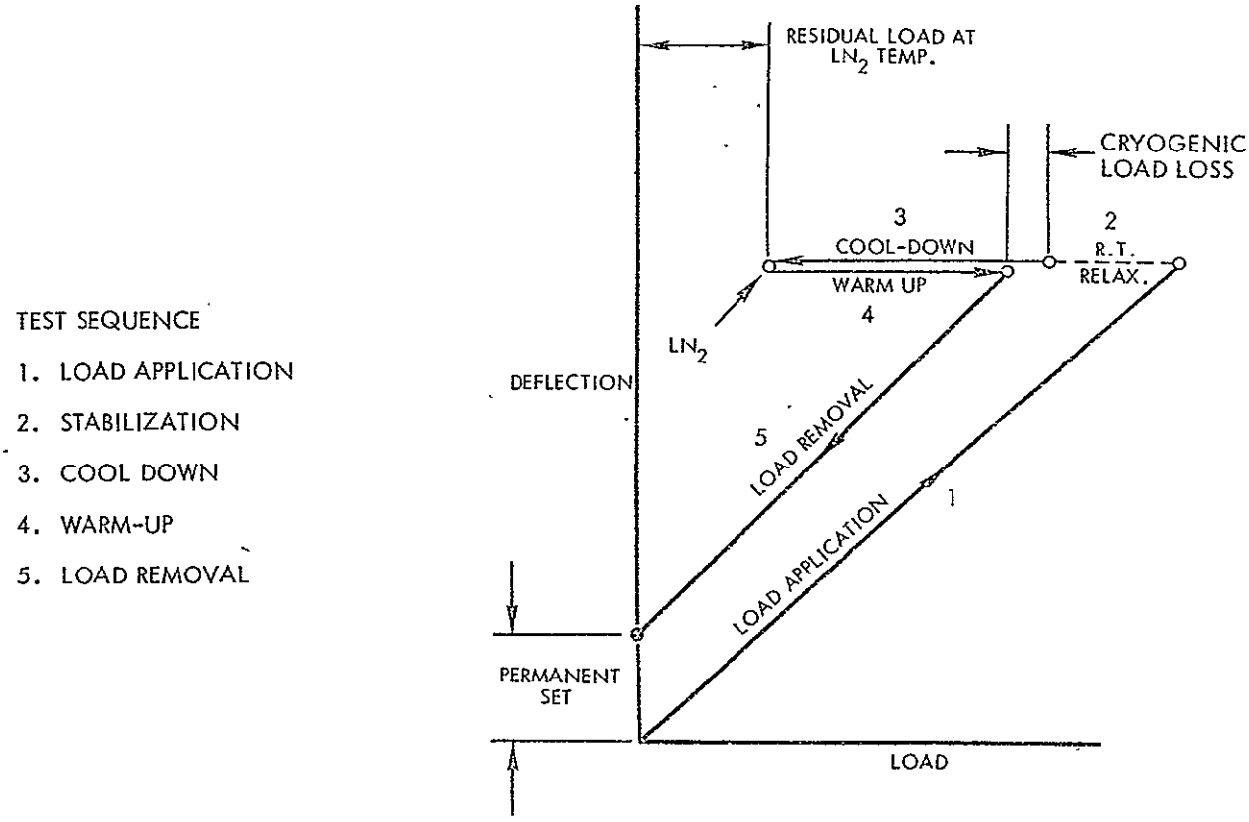

Figure 5-1. Idealized Load-Deflection Cryogenic Cycle

A schematic of the test set-up is presented in Figure 5-2. The operational procedure is as follows. After initially engaging the ram onto the sample, the reference mark is sighted, zeroed, and the load cell zero point recorded. Load is applied by torquing the ram. Load deflection data were measured at several points during the load application. When the prescribed deflection is obtained, the load required to achieve this degree of compression is recorded and the load is then allowed to stabilize at room temperature. The assembly is then cooled

to -320°F by filling the dewar flask with LN_2 to a level above that of the test specimen. After temperature stabilization, the residual load of the test sample is recorded from the load cell. The assembly is then allowed to warm-up to room temperature, the residual load recorded, then removed by untorquing the ram. The final deflection measurement is recorded at the zero load point. From this test sequence, the following data are obtained:

1. Residual load at LN_2 temperature
2. Preload loss at room temperature after a cycle from room temperature to -320°F to room temperature
3. Permanent set as a result of one temperature cycle

A compilation of significant test data is presented in Table 5-1. The compression load cycle as described above has been plotted for several representative material tests in Figures 5-3 through 5-8. The curves depict the initial ascending compression curve which is generally linear with true elastomers. After the initial load application, relaxation occurs over a period of approximately 1 hour, shown by the dotted line. During this period, there is essentially no change in deflection, only a loss in load caused by relaxation of the internal stresses. This room temperature creep was not anticipated to the degree experienced with the elastomeric materials. Plastic materials such as Teflon have a well recognized history of creep, or cold-flow, however, the percentage loss at room temperature for short term loading was no greater than that of the elastomers.

During the cool-down mode, material contraction and hardening of the test sample, as the temperature passes through the "glass transition temperature," combine to further reduce the residual stresses and result in significantly reduced load with an insignificant change in deflection. The value to which the load decays is a measure of the capability of the material to effect a seal at this low temperature. Figure 5-5 and Table 5-1 show that the polybutadiene sample relaxed to a value of only 6 psi (from approximately 200 psi) with both an initial 13 and 26 percent deflection. The control sample, Teflon, and the AF-E-71-2 shown in Figure 5-3, both maintained loads above 100 psi under these conditions.

1. APPLY PRELOAD
2. STABILIZE - R.T.
3. COOL-DOWN - LN_2
4. STABILIZE *
5. WARM TO R.T.
6. REMOVE LOAD

*RESILIENCY TO BE MEASURED BY OBTAINING LOAD-DEFLECTION DATA AT LN_2 TEMPERATURE

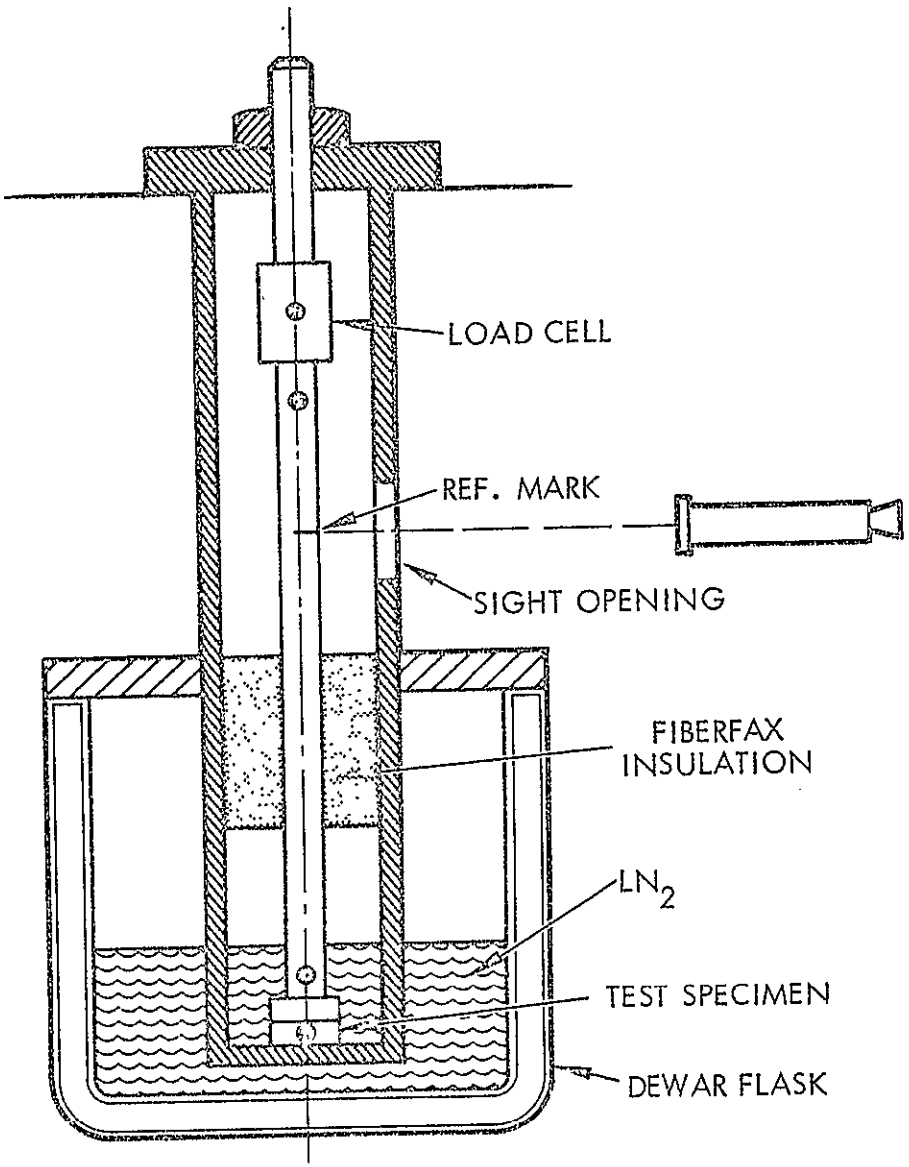

Figure 5-2. Test Fixture Schematic

Table 5-1. Compression Load Test Results

Material	Compression (%)	Final Permanent Set (%)	Loads				Load Retention		
			Initial Room Temperature (lbs)	Relax Room Temperature (lbs)	LN_2 Temperature (lbs)	Recovery Room Temperature (lbs)	Room Temperature Relaxed/Initial	LN_2 Temperature (% of Relax Load)	Room Temperature Recovery (% of Relax Load)
Viton-A (255-2)	27	2.6	284	175	67	175	62	38	100
EPT-HYSTL (AF-E-71-2)	22	1.4	592	440	142	421	74	33	96
EPT-HYDRIN (263-3)	25	3.8	577	462	128	440	80	27	96
Buna-N (215-2)	12	1.7	104	46	6	43	44	13	94
Polybutadiene (60-7)	26	1.2	269	208	6	180	78	3	86
Phenylsilicone (310-1)	24	4.2	268	226	128	134	84	57	59
Teflon (TFE)	9	4.7	606	465	153	406	77	38	88

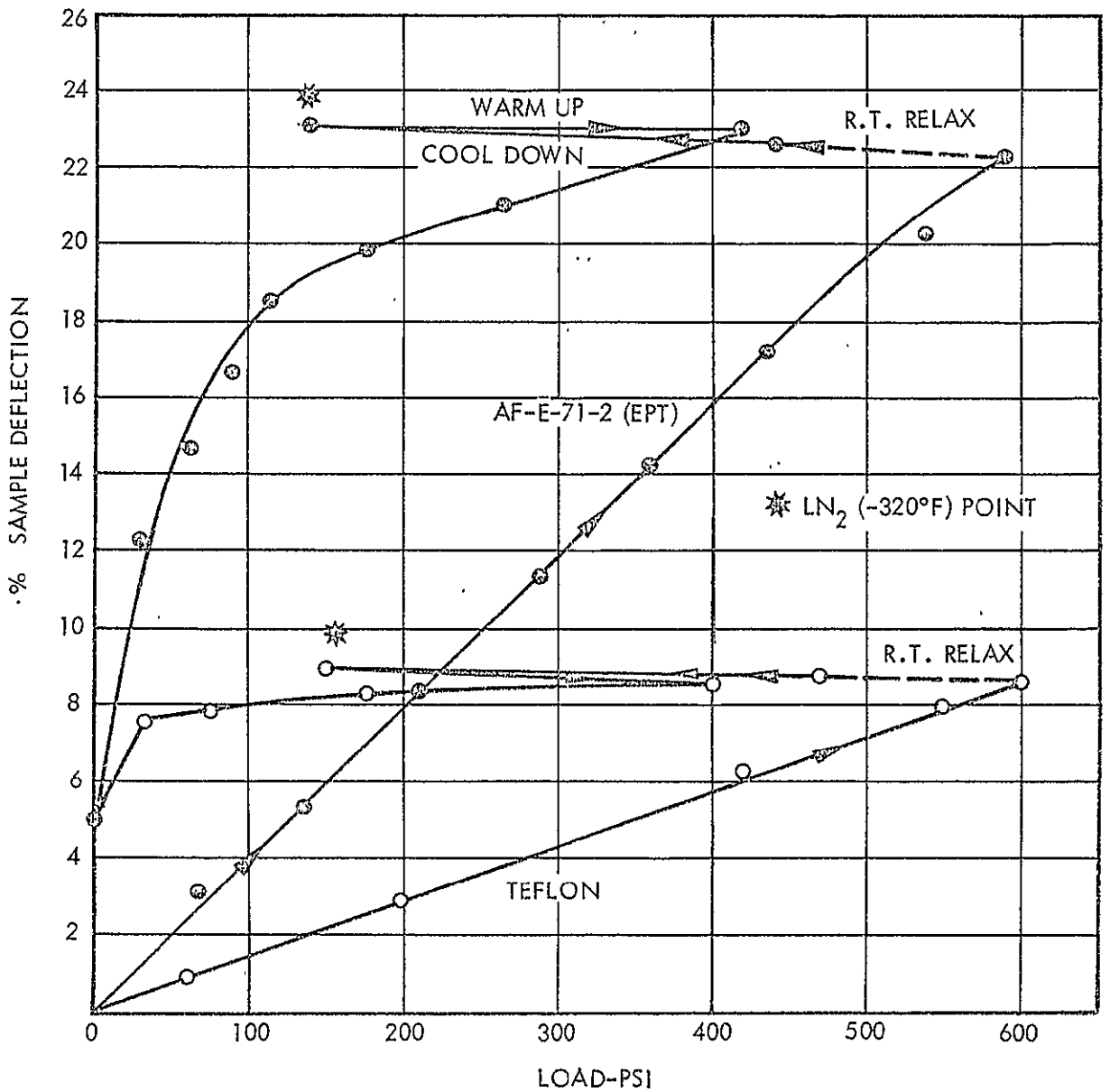

Figure 5-3. Load Deflection Curve LN_2 Temperature Cycle for AF-E-71-2 and Teflon TFE

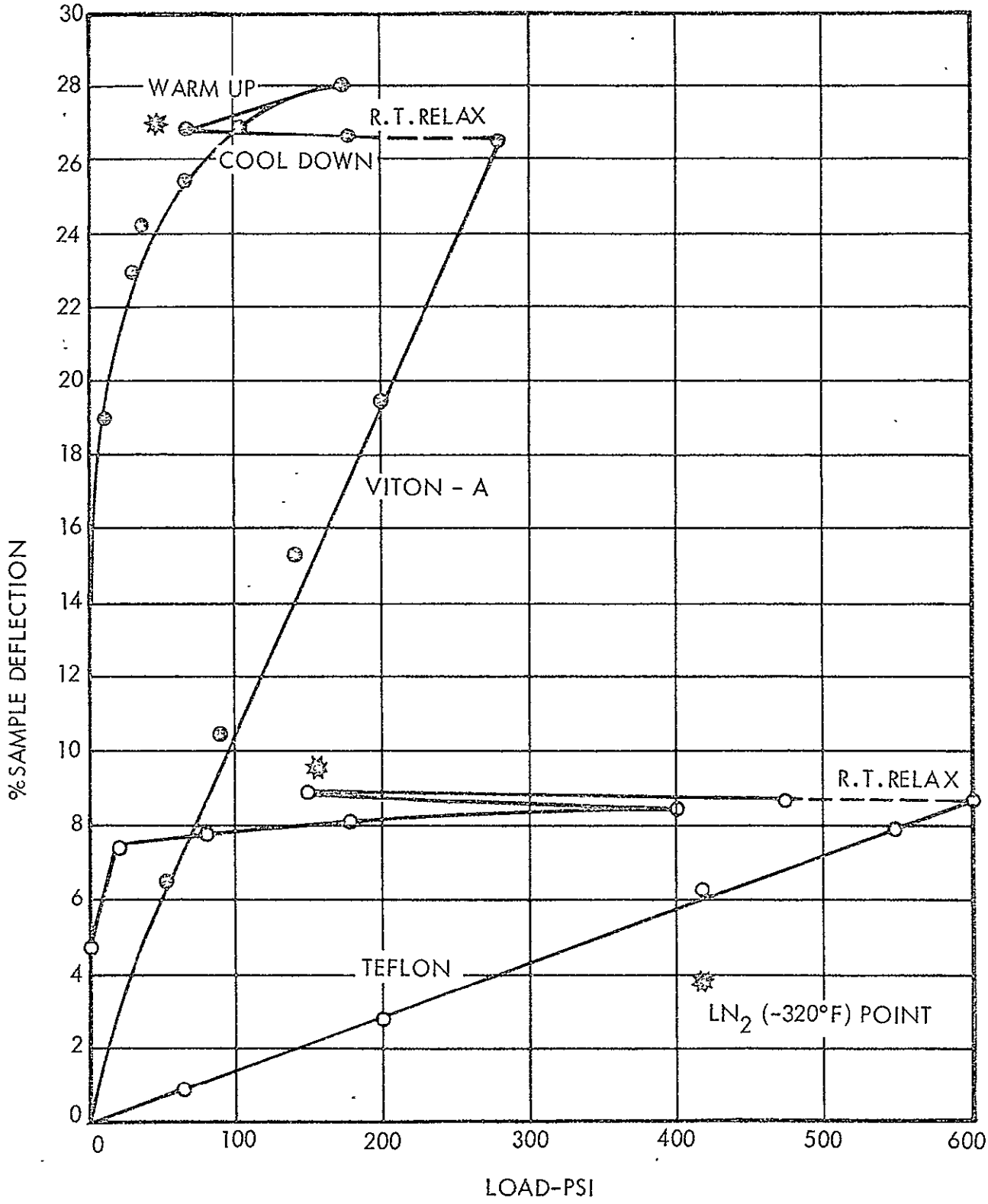

Figure 5-4. Load Deflection LN_2 Temperature Viton A and Teflon TFE

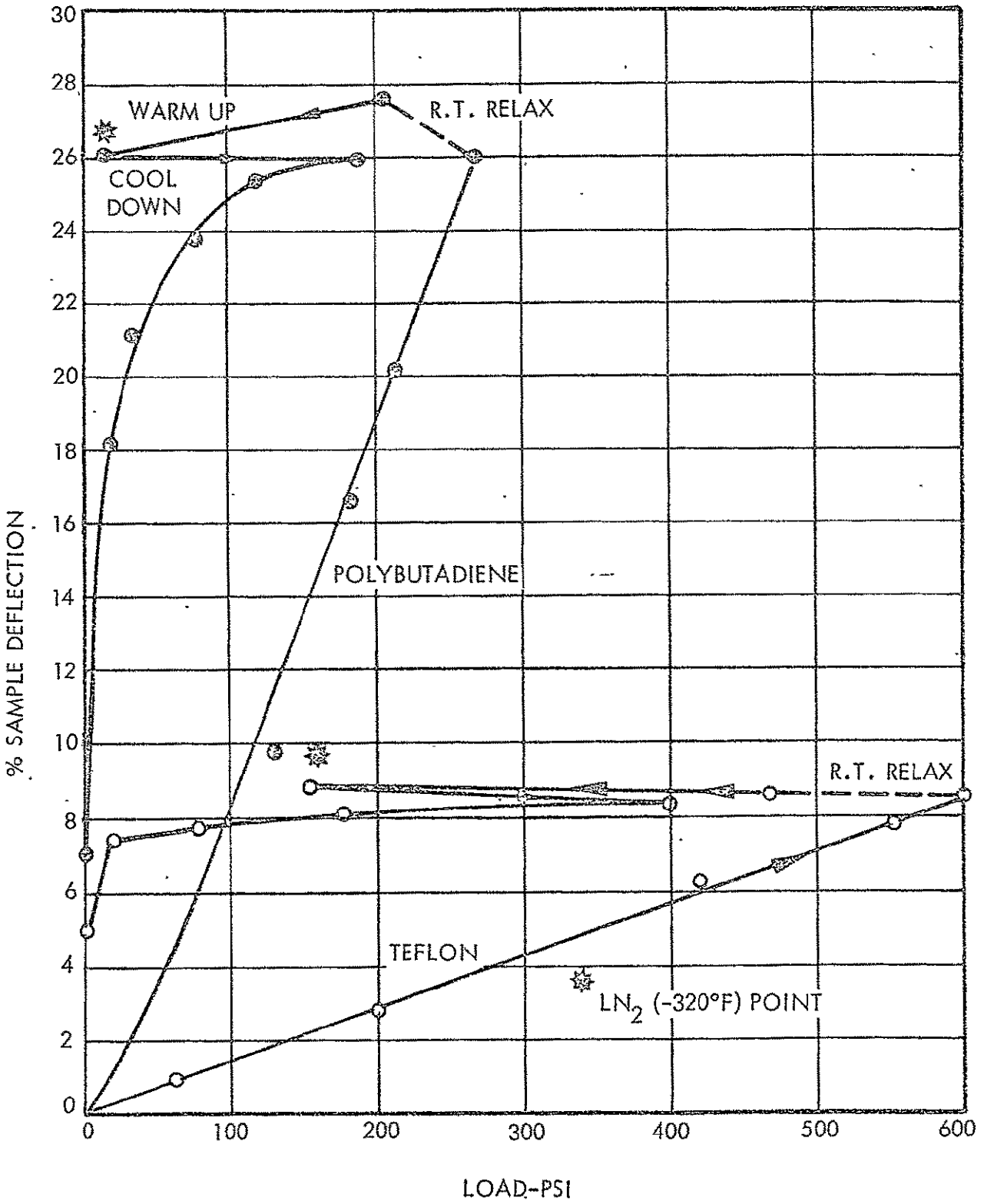

Figure 5-5. Load Deflection LN_2 Temperature Cycle Polybutadiene and Teflon TFE

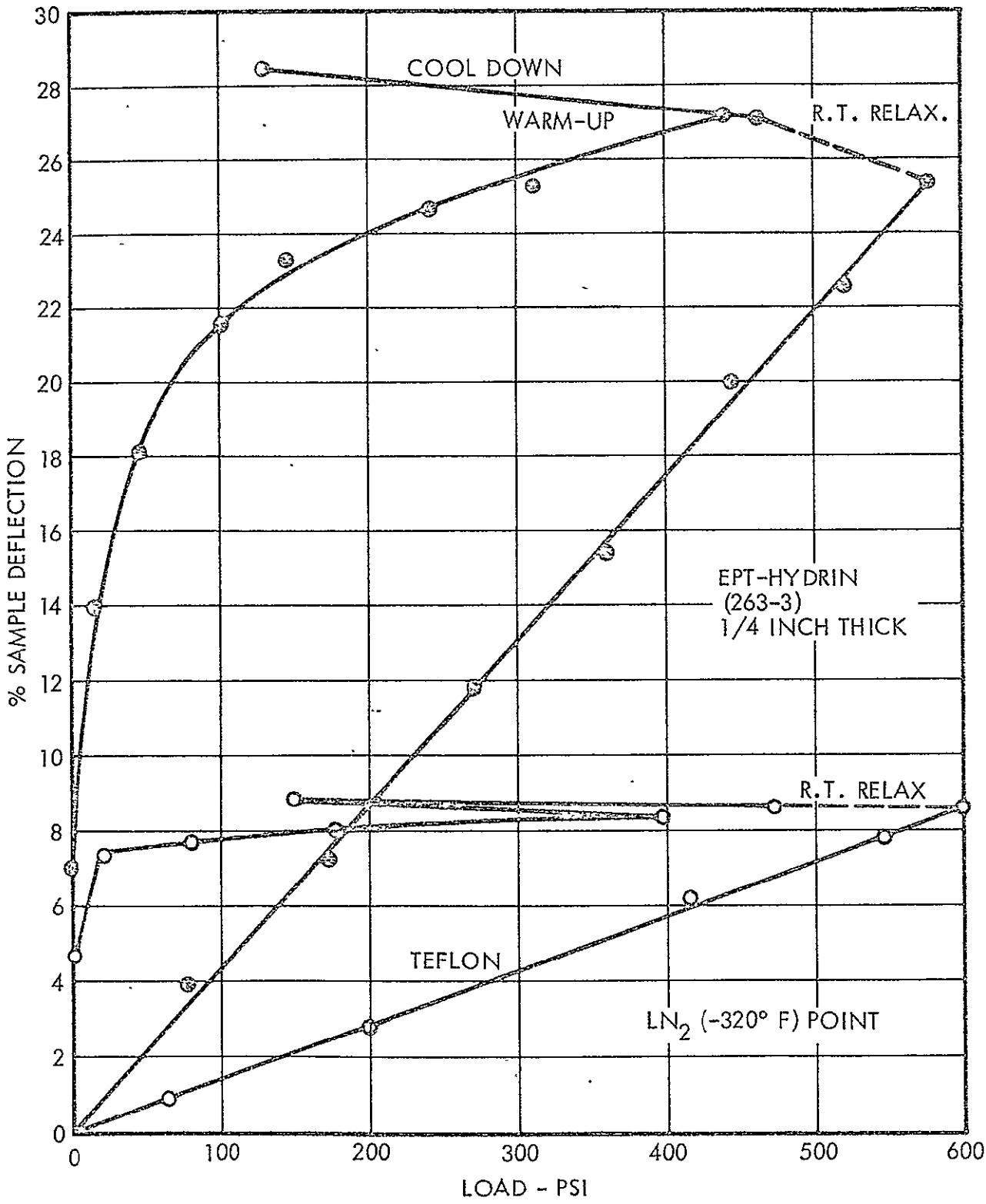

Figure 5-6. Load Deflection LN_2 Temperature
EPT-HYDRIN and Teflon TFE

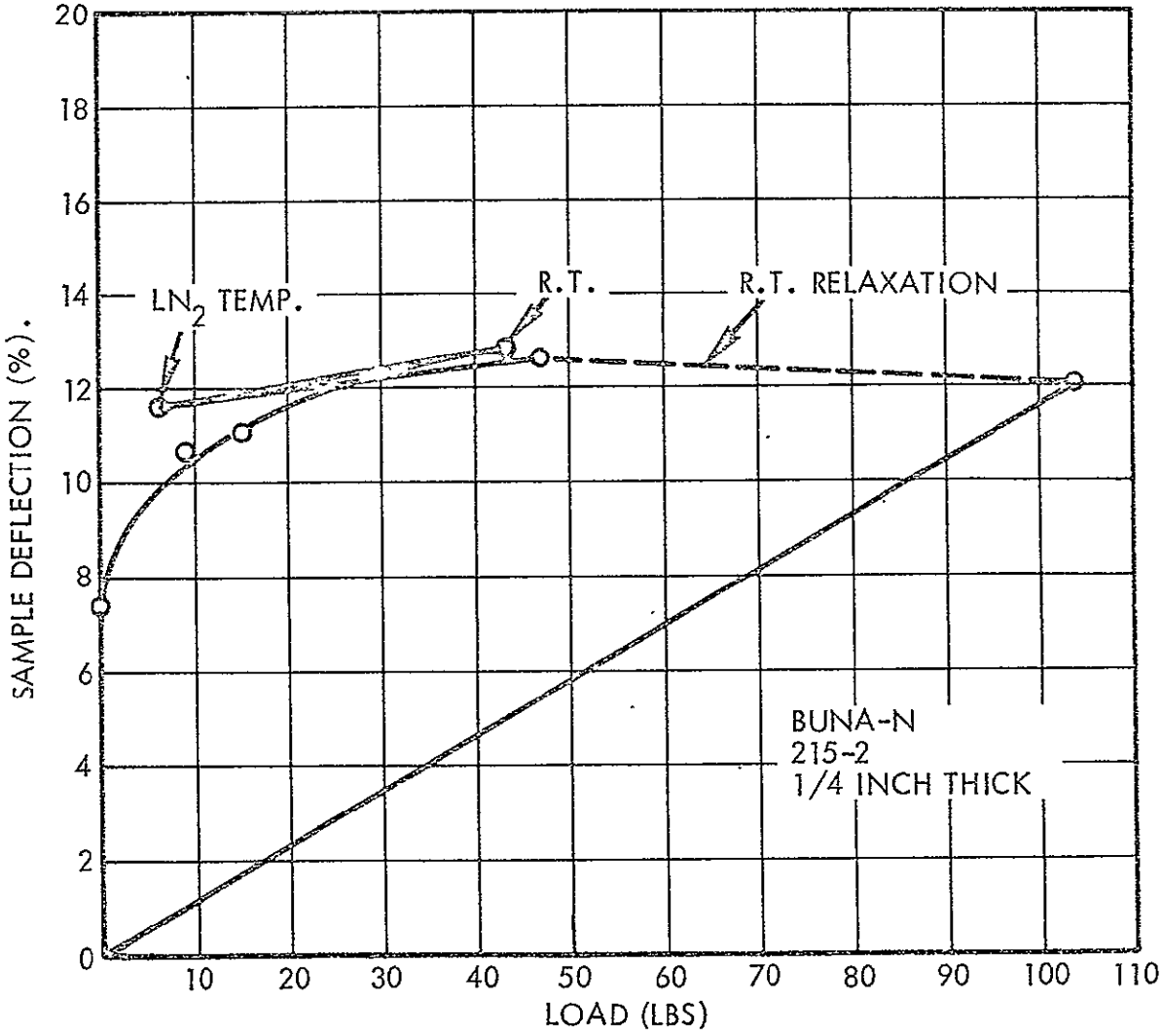

Figure 5-7. Load Deflection LN_2 Temperature BUNA N

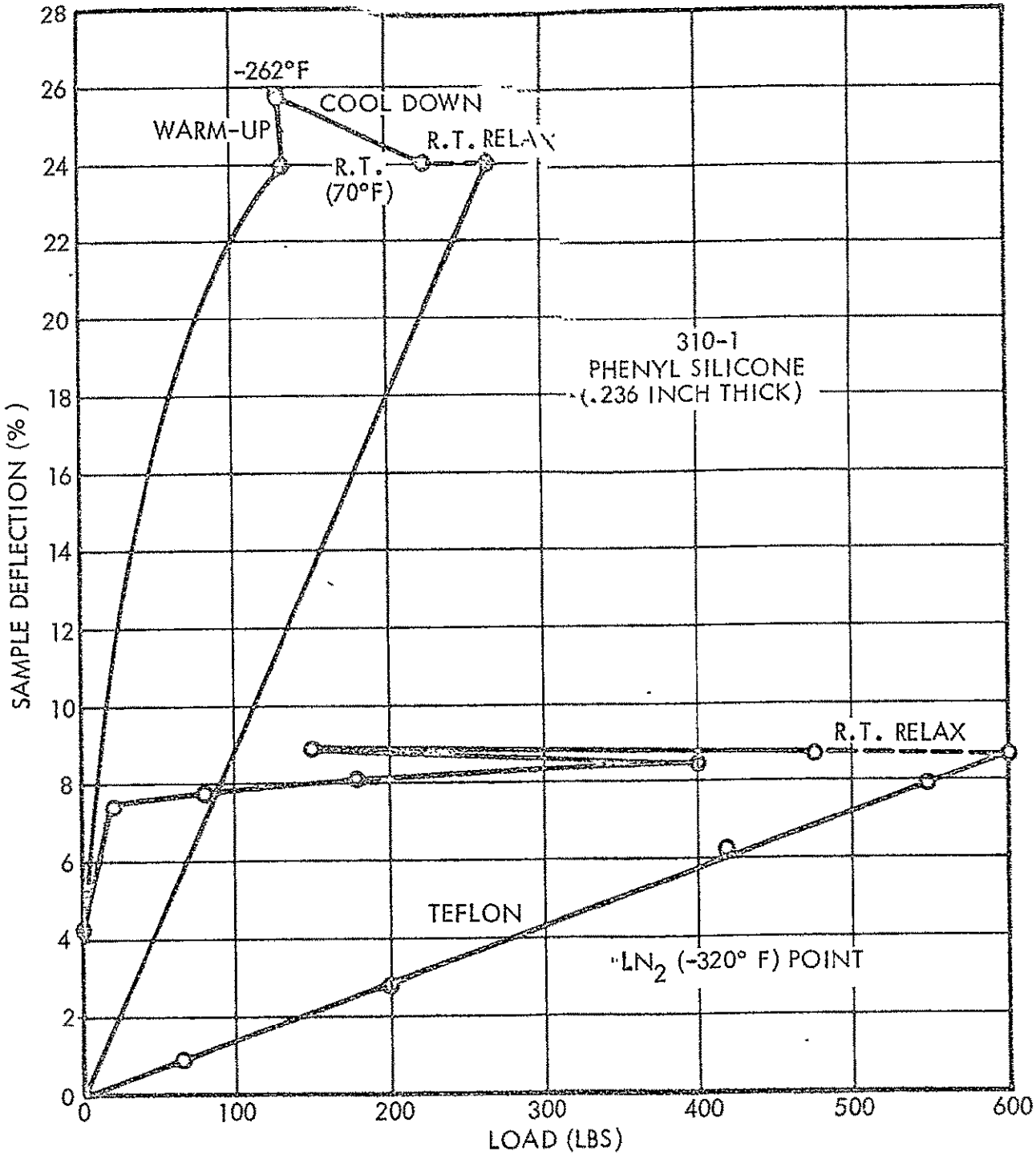

Figure 5-8. Load Deflection LN_2 Temperature Phenyl Silicone and Teflon TFE

After stabilization at LN_2 temperature, the test assembly is allowed to warm to room temperature and to again stabilize. The difference in load at this point compared to that prior to cool-down is significant in that it is a measure of the material's capability to withstand further permanent set due to temperature cycling. The AF-E-71-2, Viton A, and EPT-HYDRIN (Figures 5-3, 5-4 and 5-6) were particularly good in this regard, whereas Teflon and Polybutadiene (Figure 5-5) resulted in a significant loss of load. This step loss in load is of particular concern in applications that will be subjected to extended temperature cycling, resulting eventually in loss of sealing action even at room temperature.

During the unloading portion of the cycle, load-deflection points are recorded as the load is gradually removed. Pertinent factors are the slope of the curve and the zero load intercept. Both parameters are indications of residual elasticity and, consequently, the ability to seal. A fast drop-off in load with only minimal reduction in deflection as shown by Teflon is considered undesirable. A more gradual decay rate, as illustrated by AF-E-71-2 and Viton A in Figures 5-3 and 5-4 is more desirable. The zero load intercept is a measure of permanent set developed as a result of this one temperature cycle. The greater this value, the greater the possibility exists of losing seal effectivity with repetitive temperature cycling.

The load application, Step 1, was initially attempted at 10 percent deflection of the test sample height, initially 1/2-inch high (test buttons are 7/8-inch diameter). Preliminary results indicated, however, that the 1/2-inch sample thickness was yielding inconsistent results caused by excessive unequal cooling rates. Satisfactory results were obtained when the thickness was reduced to 1/4 inch. Difficulty was also encountered with the 10 percent initial deflection. Excessive load loss was experienced on cool-down because of the relatively light load obtainable with elastomers at this compression setting.

After completion of the first series of screening tests, the test set-up was modified in an attempt to improve the transient temperature lag during cool-down and warm-up. It had been anticipated that a determination of the Tg of the test sample could be obtained by detecting the change

in contraction rate and elasticity of the polymer during the temperature transient. However, this data had not been obtainable with the original test set-up due to differences in thermal conduction and contraction rates between the stainless steel fixture and the test sample. It was also apparent that the intermediate temperature points would be difficult to obtain with the existing test set-up. The test modification consisted of installing the entire compression test fixture in an environmental chamber, (Figure 5-9). This permitted a more accurate control over the ambient temperature surrounding the fixture during cool-down, resulting in uniform cooling of the structure and test sample. Load cycling was achieved by use of pneumatic loader located at the top of the test fixture. Direct load measurements were obtained from a load cell and displacement measurements by use of a LVDT position transducer located across the test specimen. Continuous readout of three temperatures (test sample, fixture and chamber), load and deflection were obtained on a 6-channel recorder. In addition, load versus temperature was recorded on a X-Y plotter. The test procedure sequence was also modified to incorporate stepped temperature changes of approximately 25°F. After stabilization at each temperature, the load on the test sample was cycled to obtain load/deflection resiliency data.

The new test sequence permitted the simultaneous determination of the percentage loss in initial applied load and load deformation data with decreasing temperature. A representative curve of the type of data being obtained is shown in Figure 5-10. Illustrated are two TRW compounds, AF-E-71-2, an EPT base polymer developed under AF Contract F33615-69-C-1535 specifically for hydrazine service, and 310-1, a silicone base polymer, specially compounded to improve the physical properties of this base material. As will be noted, the latter material has maintained the good low temperature load deformation properties expected of silicone polymers.

Considerable effort was expended testing these two materials in attempting to improve the techniques required to establish a repeatable test sequence in order to detect the extremely small deflections caused by load changes inherent with elastomers at cryogenic temperatures.

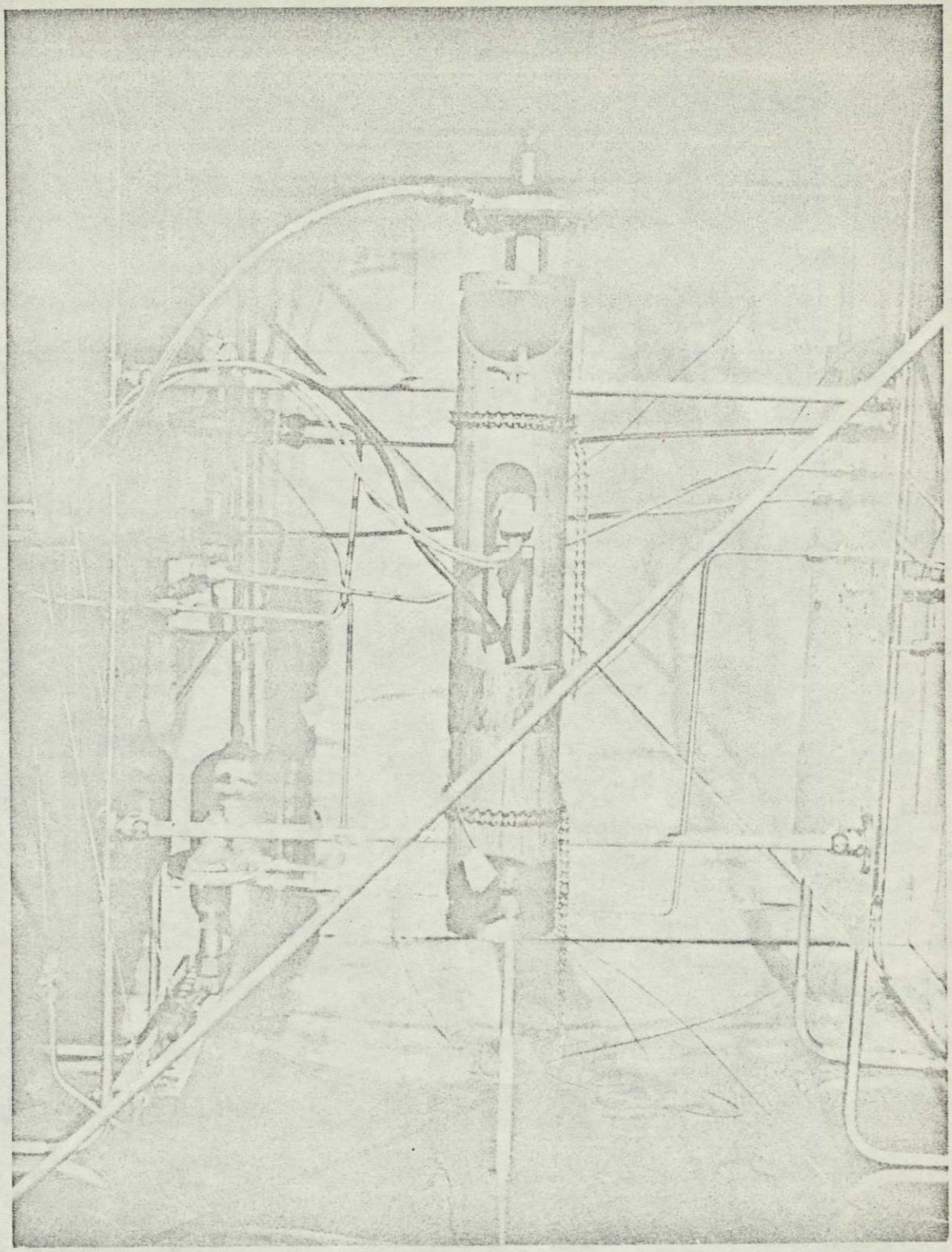

Figure 5-9. Seal Material Evaluation Test Fixture
(Installed in Cryogenic Test Chamber)

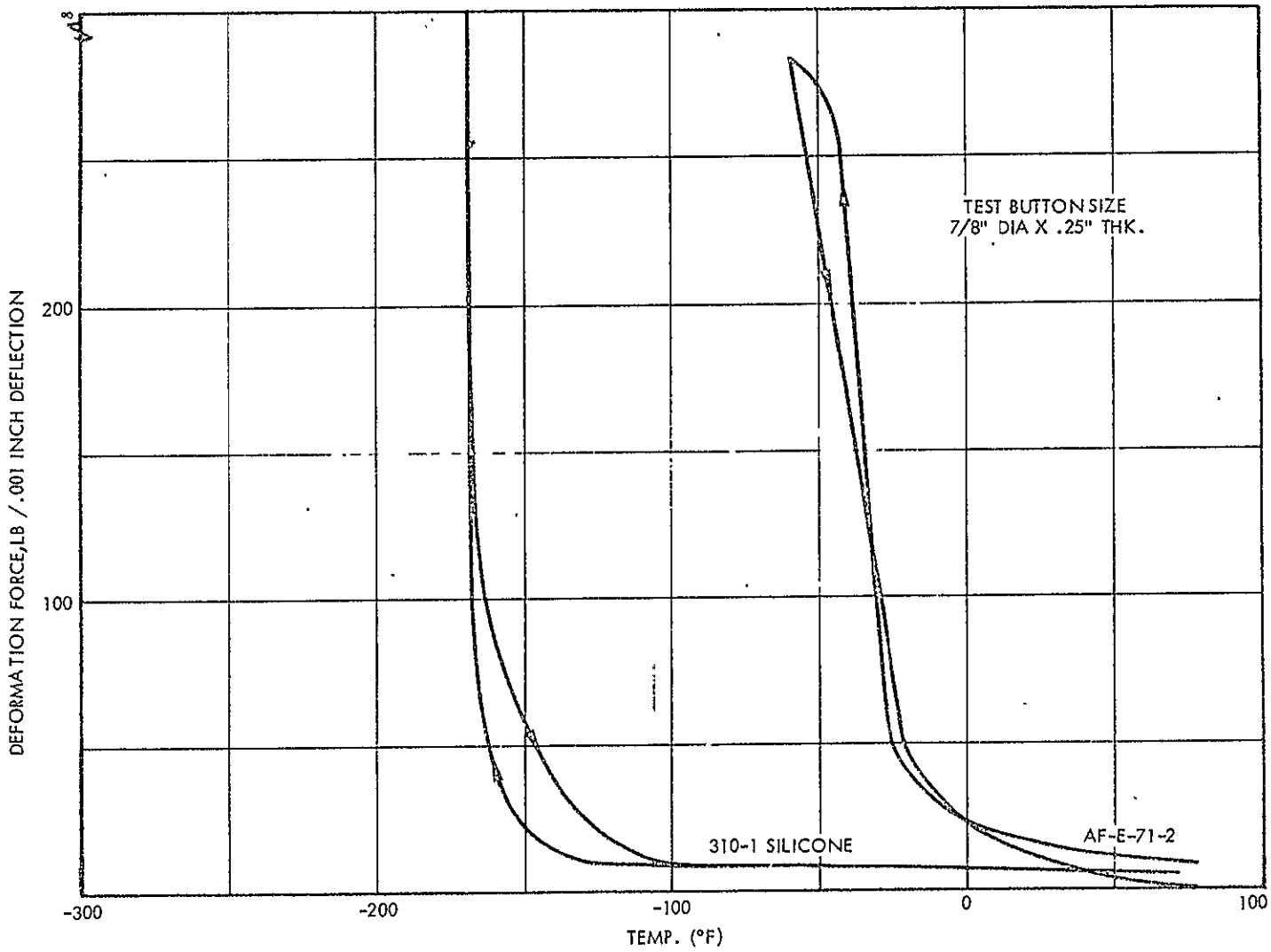

Figure 5-10. Deformation Force Versus Temperature

This effort, however, was unsuccessful; no additional information was derived over that originally obtained from the compression load tests and the test series was discontinued. The remainder of the LN_2 evaluation tests were devoted to obtaining comparative performance data with the selected materials installed in a valve.

5.1.2 LN_2 Valve Seal Tests

The valve seal tests were added to the screening portion of the program to provide more detailed quantitative data with which to base the selection of materials for the following LH_2 test program. The minute deflections required to effect a seal at cryogenic temperatures could not be measured directly as attempted during the compression load tests because of the relatively large errors generated by the fixture and the sensitivity of the instrumentation when subjected to gross temperature changes. The use of gas leakage rates as an indication of seal deflections greatly simplified the task of obtaining comparative deflection data for the individual test samples. Quantitative measurement of gas leakage rates by the water displacement method proved to be both reliable and repeatable. Calibration of the valve dome loader at various temperatures throughout the operation range indicated only a minimal shift with temperature. To permit utilization of the dome loader at cryogenic temperatures, the elastomeric diaphragm was replaced with one made from 316 stainless steel. The pressure force curve proved to be linear throughout the applied pressure range of 0 to 100 psig (Figure 5-11). Development of the testing techniques, hardware and instrumentation that would be used later in the LH_2 valve test series were developed economically using LN_2 testing facilities.

The LN_2 valve test series was designed to directly measure sealing characteristics of the selected materials by measuring the load required to effect a seal against helium at LN_2 temperatures ($-320^{\circ}F$). Helium was selected as the pressurant gas because of its low liquefaction temperatures, ease of handling and low density which makes it difficult to seal. Initially, helium was also selected because it would permit the utilization of a mass spectrometer halogen leak detector; however, this proved to be excessively time consuming for this program. Residual gas in the outlet

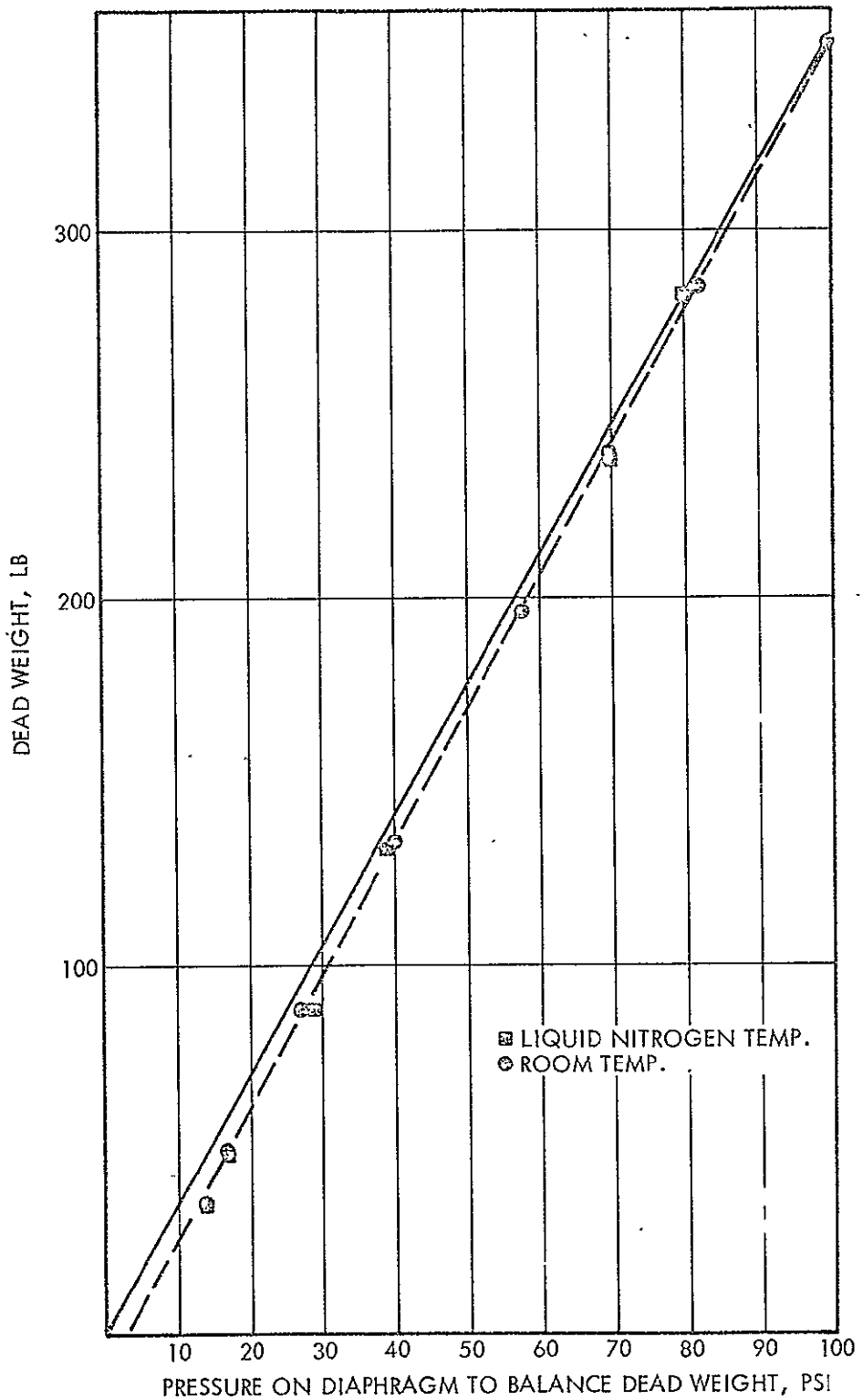

Figure 5-11. Calibration of Diaphragm Load on Valve Test Apparatus

line consistently resulted in erroneous leak indications. Quantitative measurements were particularly unreliable. The use of a simple water displacement leak indication proved to be satisfactory and expedient throughout the test series. Although problems were also anticipated with erroneous leak indications caused by thermal expansion of the cold gas trapped in the outlet line after valve shutoff, this proved to be easily controlled. A coil of 1/4-inch diameter tubing was submerged in a room temperature water bath and close coupled to the valve outlet thus minimizing the volume of cold gas that would be subjected to warming. Insulation jacketing of the exposed line between the water bath and the valve outlet was found to be unnecessary. Throughout the test series, positive zero leakage was repeatably demonstrated, nor was there any indication of cryogenic pumping (the reverse flow of gas into the valve caused by cooling contraction of the gases in the lines).

The valve, schematically portrayed in Figure 5-12, was installed in a dewar and submerged in LN_2. After temperature stabilization, helium pressure was applied to the valve inlet and verification of flow was obtained. Helium pressure was then applied to the dome actuation pressure port and gradually increased until the flow stopped. Valve inlet pressures up to 400 psig were tested. Sealing characteristics at intermediate temperatures of -110° and -200°F were also checked on some materials. During the cool-down periods, the valve was left in the open position to preclude the possibility of the seal conforming to the seat at room temperature and subsequently exactly mating with the seat after hardening at cryogenic temperatures. Tests were performed with the seat loaded during cool-down and consistently yielded good sealing properties as contrasted with the results obtained when the valve was open during cool-down.

After the initial sealing cycle, the valve was cycled an additional six times by alternately venting and pressurizing the dome loader with 400 psi at the valve inlet. The actuator dome pressure was then gradually increased until flow through the valve stopped or the maximum dome loader pressure was reached. Some of the materials tested, although capable of achieving a seal on the initial load application, were unable to seal after the actuation cycles. The test samples were cut from sheet stock 0.070 to 0.100 inch thick and were approximately 0.25 inch in diameter.

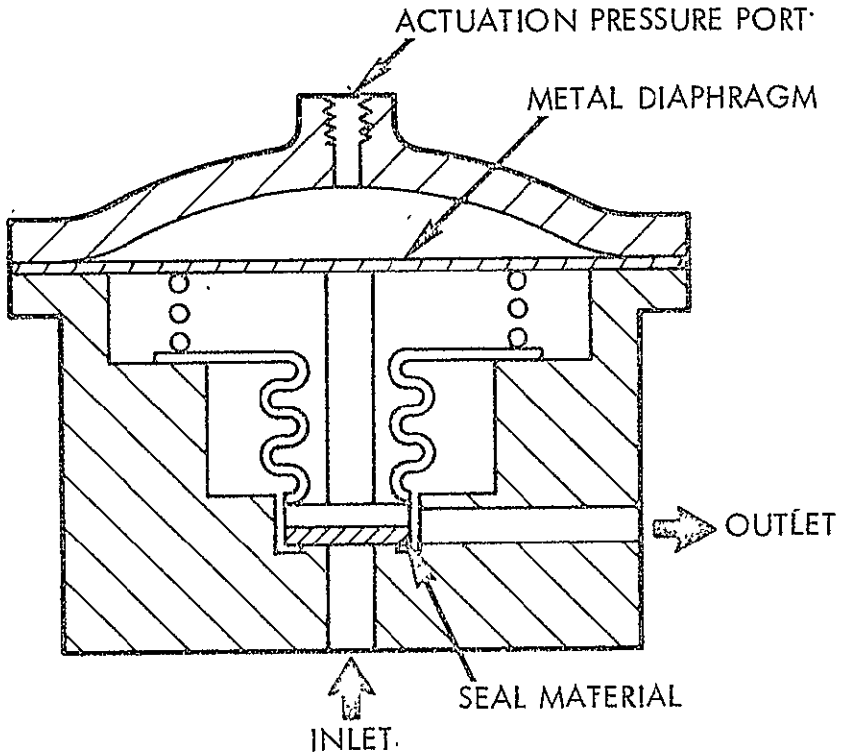

Figure 5-12. Schematic Poppet Seal Test Fixture

The initial installation of the seal was accomplished by attaching the seal to the poppet by means of a screw through the center. This was subsequently found to be both undesirable and unnecessary. The close fit of the poppet and seal in the bore, coupled with the direction of flow sufficed to hold the seal in place. Also, the attachment screw was found to be applying unequal pre-stress to the elastomer and attempts to apply uniform torquing to individual materials of varying hardness was impractical. With the exception of AF-E-124D, all materials had a surface finish of between 8- and 16 microinches. The AF-E-124D, because of its inherent critical molding process, had an irregular finish. A qualitative comparison of the effects of finish and elasticity shows the difference in dome pressures required to effect a seal at room temperature. The finish of the valve seat was 32 microinches. No special processes were used to improve the finish over that received; in effect, a typical commercial valve seat was used throughout the test series.

5.1.3 LN_2 Test Results

The LN_2 compression load tests were summarily successful in several respects. Immediate confirmation was established regarding the capability of certain elastomers to withstand cryogenic environments under high load, a relatively simple test procedure was established as an acceptable means for obtaining comparative low temperature material properties, and significant material properties most pertinent to good cryogenic operation were identified.

In the past, the use of elastomers in cryogenic applications has been generally ignored because of the belief that all polymeric compounds become brittle as they harden at temperatures below their brittle point or Tg· thus, the belief that they would be unserviceable for sealing applications which inherently involve high concentrated stress loading. The compression load tests succeeded in identifying several polymers capable of sustaining high loads, including repetitive cycling.

During the program, those materials identified in the proposal and selected during task one of this program were evaluated. The materials subjected to the load compression tests were:

Viton-A
AF-E-71-2
Buna-N
Polybutadiene
EPT-HYDRIN
Phenyl Silicone
Teflon TFE

Load-deflection curves for these materials are presented in Figures 5-4 through 5-8. The Teflon curve has been added to each curve for direct comparison purposes.

As mentioned earlier, the pertinent factors to be studied were the residual preload or load retention at -320°F, the percentage recovery of load after the subsequent warm-up to room temperature, and the permanent set resulting after removal of load.

A summary of the pertinent data is presented in Table 5-1. An analysis of these data resulted in the following classifications being assigned to these materials regarding additional testing.

Acceptable	Unacceptable
Viton-A	Buna-N
AF-E-71-2	Polybutadiene
EPT-HYDRIN	
Phenyl Silicone	

As can be noted from the data, the first three "acceptable" materials maintain 96 to 100 percent of the initial relaxed preload after temperature cycled to -320°F as compared with 88 percent for Teflon. The phenyl silicone sample, although only maintaining 59 percent of the relaxed preload after the temperature cycle, was capable of maintaining 57 percent of this load at -320°F, indicative of a high probability that this material could prove useful as a static seal regardless of the low load recovery of only 59 percent at room temperature. This would be

indicative of a need for additional initial preload to compensate for this deficiency. Because of the low retained loads at LN_2 temperature observed for both the Buna-N and polybutadiene samples, these were dropped from further consideration in the program. Neither material is capable of high temperature operation, and they are LO_2 impact sensitive.

The LN_2 valve tests were conducted with the four materials selected from the compression load tests and four other materials that had previously been selected for evaluation, but which had not been available during the initial test series. These new materials were fluorosilicone, polyethylene, carboxynitroso and HYSTL. In addition, a polybutadiene sample was evaluated to provide correlation data with that of the previous test series. The compression seat loads required to effect a seal were recorded at room temperature, -109° and -320°F. Table 5-2 summarizes the data by listing recorded leakage at various valve inlet pressures and temperatures. The fact that sealing action through seal deformation was achieved was indicated by the gradual reduction in rate of leakage as the dome pressure was increased. This adds validity to the test results for use in comparing the relative performance between materials. Higher loads were also required as the operating temperature was lowered between -109° and -320°F. A comparison of the sealing loads required at LN_2 temperatures and those obtained later with LH_2 (-423°F) do not materially differ, substantiating the theory proposed at the start of the program that only a minimal change in material properties would be expected through the range of temperatures below the Tg. A comparative evaluation of the data resulted in classification of the tested materials as follows:

Good	Fair	Poor
Teflon	Phenyl Silicone	Polybutadiene
AF-E-71-2	Viton-A	Fluorosilicone
HYSTL		Polyethylene
		Carboxynitroso (Nitroso)

Table 5-2. LN_2 Valve Seat Test Result Summary

LEAKAGE PERFORMANCE

Material	Temp °F	Pressure (psi)	Seat Stress (psi)	Bubbles per Minute: 0 Zero	<10 Slight	10 > 50 Moderate	>50 Gross
Teflon TFE	70	400	500	---			
Control Material	-109	400	5000	---			
	-320	10	4900	---			
	-320	200	4900	---	---	---	
	-320	400	5500	---			
	-320	400	5500	———	———	———	
	-320	400	6200	———			
Selected Materials							
AF-E-71-2	70	400	300	---			
EPT	-109	400	3000	---			
	-320	10	2600	---	---	---	---
	-320	10	3000	---			
	-320	100	3000	---	---	---	---
	-320	400	3600	---			
	-320	400	3600	———	———	———	———
	-320	400	4300	———	———	———	———
	-320	400	4900	———			
60-7	70	10	300	---			
Polybutadiene	70	400	300	---			
	-109	400	300	---	---		
	-109	400	500	---			
	-320	10	500	---	---	---	---
	-320	10	1400	---	---	---	---
	-320	10	5600	---	---	---	---
	-320	10	6200	---	---		
	-320	400	6200	---	---	---	---
	-320	400	6900	---	---	---	---
	-320	400	7500	---	---	---	
	70	400	500	following low temperature tests			
316-1	70	40	300	---	---	---	
Fluorosilicone	70	80	500	---	---	---	---
	70	120	800	---	---	---	---
	70	400	1100	---			
	-109	400	1100	---			
	-320	25	7500	---	---	---	---
	warmed and cooled at 1100 psi seat stress—sample tore						
310-1 First Sample	70	400	300	---			
Phenyl Silicone	-109	400	300	---			
	-320	50	7500	---	---	---	---
	warmed and cooled at 6200 psi seat stress						
	-320	50	6200	---	---	---	
	-320	100	6200	---	---	---	
	-320	150	6200	---	---	---	---
	-320	400	7500	---			
	-320	100	7500	———	———	———	
	-320	150	7500	———			———
310-1 Second Sample	75	80	1100	---	---	---	---
	75	400	1400	---			
	cooled closed at 1100 psi seat stress						
	-320	400	6200	---			
	warmed and cooled open						
	-320	50	6200	---	---	---	
		400	6200	---	---	---	---
255-2	70	400	300	---			
Viton A	-109	10	300	---	---	---	---
	-109	10	3000	---			
	-109	100	3000	---			
	-109	10	7500	———	———		
	-320	10	7500	---	---	---	---
Other Materials							
Polyethylene	20	400	2300	---			
	-109	400	2600	---			
	-320	10	7500	---	---	---	---
299-1 Nitroso	70	400	500	---			
	-109	400	1100	---			
	-320	10	7500	---	---	---	
	-320	100	7500	---	---	---	---
HYSTL (TRW)	70	400	300	---			
	-109	400	300	---			
	-320	400	500	---			

--- before cycling

——— after cycling open and closed 6 times following temperature reduction

Materials rated "poor" were dropped from the list of candidate polymers except for the fluorosilicone, while served as a control for the phenyl silicone sample. A summary of the results is presented in Table 5-3.

Table 5-3. Seat Loads and Leakage at LN_2 Temperature

Material	Seat Load Required to Seal (psi)	Leakage Before Cycling (Bubbles/Min)	Leakage After Cycling
AF-E-71-2	4900	0	0
Viton-A	7500	>50	*
Phenyl Silicone	6200	0	>50
Polybutadiene	7500	>10	*
Fluorosilicone	7500	>50	*
Polyethelene	7500	>50	*
Nitroso	7500	>50	*
HYSTL	300	0	0
Teflon	6900	0	0

*Did not cycle because of high initial leakage rate

The magnitude of the loads required to effect a seal during this test series was in general significantly higher than anticipated, and also higher than that recorded during the LH_2 valve test series where leakage was also checked with the test assembly stabilized at -320°F. However, the relative ranking of the respective materials was consistent. The Viton-A and AF-E-71-2 samples are noteworthy exceptions. Both materials were those selected for the first series of test during which period testing techniques were still being developed.

5.2 LO_2 TESTS

The LO_2 test program was intended to serve as an additional screening series for those materials from the initial group selected for evaluation during Task I to identify those compounds not compatible with LO_2. Several of the selected compounds were new, for which no prior data were available or were minimal, regarding LO_2 service. The tests

were performed simultaneously with the LN_2 evaluation tests and consisted of a 26-day soak test in LO_2 maintained continuously at LN_2 temperature of -320°F. Two to four physical test specimens of each material (standard tensile test "dogbone" configuration, 1-1/2 inches long) were immersed in individually sealed 20 ml aliquots containing LO_2. The aliquots were submerged in LN_2 to maintain the temperature at -320°F throughout the test period.

The materials tested were:

- AF-E-71-2
- Polybutadiene
- EPT-HYDRIN
- Viton-A
- Buna-N (Nitrile)

The test procedure for the LO_2 compatibility soak tests follows:

1) Submerge two samples in LO_2 in clean glass flask and then seal.

2) Hold for 26 days at LO_2 temperature.

3) Allow sample flask to warm to room temperature trapping gases as they evolve.

4) Analyze gas samples and any residue in flask.

5) Subject material test samples to hardness test followed by tensile pull test. Two control samples of each material batch which have not been subjected to LO_2 exposure shall be simultaneously tested for comparison of results.

After storage, the LO_2 was transferred to a stainless steel container and the gaseous oxygen subsequently analyzed by infrared spectrometry for volatile impurities that may have evolved from the test samples. No infrared active impurities were detected.

Samples were weighed before and after exposure. Weight changes, as reported in Table 5-4 were minimal. An examination of the test containers and the samples after completion of the exposure period did not reveal the presence of any nonvolatile impurities.

Table 5-4. Weight Changes After LO_2 Exposure

26 days at -320°F

Sample Weight, grams

Sample Identification	Initial	After LO_2 Storage	Change
Buna-N			
1	0.4901	0.4907	+0.0006
2	0.5389	0.5395	+0.0006
3	0.4384	0.4391	+0.0007
Polybutadiene			
1	0.5316	0.5318	+0.0002
2	0.5355	0.5304	+0.0009
3	0.3604	0.3599	-0.0005
EPT-HYDRIN			
1	0.4947	0.4956	+0.0009
2	0.5430	0.5452	+0.0022
3	0.4680	0.4645	-0.0035
AF-E-71-2			
1	0.4020	0.4019	-0.0001
2	0.4681	0.4679	-0.0002
3	0.3710	0.3711	+0.0001
4	0.3397	0.3399	+0.0002
Viton-A			
1	0.8662	0.8671	+0.0009
2	0.8444	0.8452	+0.0008
HYSTL			
1	0.9005	0.9006	+0.0001
2	0.9246	0.9246	-0.0001
3	1.0340	1.0340	None
Phenyl Silicone			
1	0.6939	0.6952	+0.0013
2	0.6845	0.6857	+0.0013
3	0.6957	0.6971	+0.0014
Fluorosilicone			
1	0.9278	0.9287	+0.0009
2	0.102	0.9111	+0.0009
3	0.9189	0.9208	+0.0019

Hardness, elongation and tensile tests were conducted on all test samples together with identical samples which had not been exposed to the LO_2 soak tests. The results of these tests are presented in Table 5-5.

Three additional sample specimens which had subsequently been added to the program were also subjected to the LO_2 soak test for a similar period of 26 days. These materials were HYSTL, phenyl silicone and fluorosilicone. As with the original group tested, no appreciable weight or dimensional changes could be detected. As a consequence, the physical property tests were not conducted on these last specimens. The results of the weight measurements are presented in Table 5-4.

There was no evidence of deterioration, discoloration, or change in physical properties with any of the materials tested. No materials were eliminated from the program as a result of this test series. All the materials except the buna-N and the polybutadiene, which were later dropped because of inferior results in the LN_2 compression load and valve tests were selected for the LO_2 ABMA impact test series.

Table 5-5. LO_2 Compatibility Test Results

	Tensile (psi)	Elongation (%)	Hardness (Shore A)
60-7, Polybutadiene			
*Control	1400	440	75
**Exposed	1525	575	75
215-2, Nitrile			
Control	2500	325	70
Exposed	2400	300	71
255-2, Viton-A			
Control	2250	160	80
Exposed	2200	160	80
263-3, EPT/HYDRIN			
Control	1400	130	85
Exposed	1200	120	80
AF-E-71-2			
Control	1800	110	90
Exposed	1800	100	81

*Control: Samples from same batch as those subjected to LO_2 exposure.

**Samples subjected to 26 days continuous exposure to LO_2 at LN_2 temperature.

5.2.1 LO_2 Impact Tests

LO_2 impact sensitivity tests were conducted in accordance with standard ABMA test requirements using a 20-lb weight dropped from a height of 43.3 inches, resulting in an impact energy of 72 ft-lb. All test apparatus was thoroughly LO_2 cleaned and checked with ultraviolet light for hydrocarbon impurities. The test samples were sonic cleaned in isopropyl alcohol and then vacuum dried for 2 hours at 200°F prior to testing. LN_2 was used as the coolant. A photograph of the impact test equipment is shown in Figure 5-13. Proper operation of the guillotine was verified by measuring the drop time of the weight and comparing it with the calculated theoretical drop time. Where reactions were observed at the standard height, additional tests were performed at progressively lower impact loads to determine the impact sensitivity level of each material. The respective impact parameter for each height tested is listed below.

Impact Energy (ft-lb)	Drop Height (in.)	Drop Time (msec)		Difference* (percent)
		Calculated	Actual	
72	43.3	474	475	0.2
60	36.0	432	434	0.5
50	30.0	394	397	0.7
40	24.2	353	360	0.9
30	18.1	314	316	0.6
20	12.0	249	252	1.2

*Allowable variation of actual to calculated drop times is ±3 percent.

Three impact sensitivity test series were conducted during the ABMA impact test program. The results of the first series is reported in Table 5-6. The second series was discontinued because all test samples flashed upon impact, including the Teflon samples.

An investigation showed that the cleaning procedure for the samples had not been properly conducted, and consequently, either original impurities or inadequate vacuum drying subsequent to cleaning had caused the problem. Additional investigation of the effects of different types of cleaning solvents and the development of adequate vacuum drying procedures for production service applications is recommended.

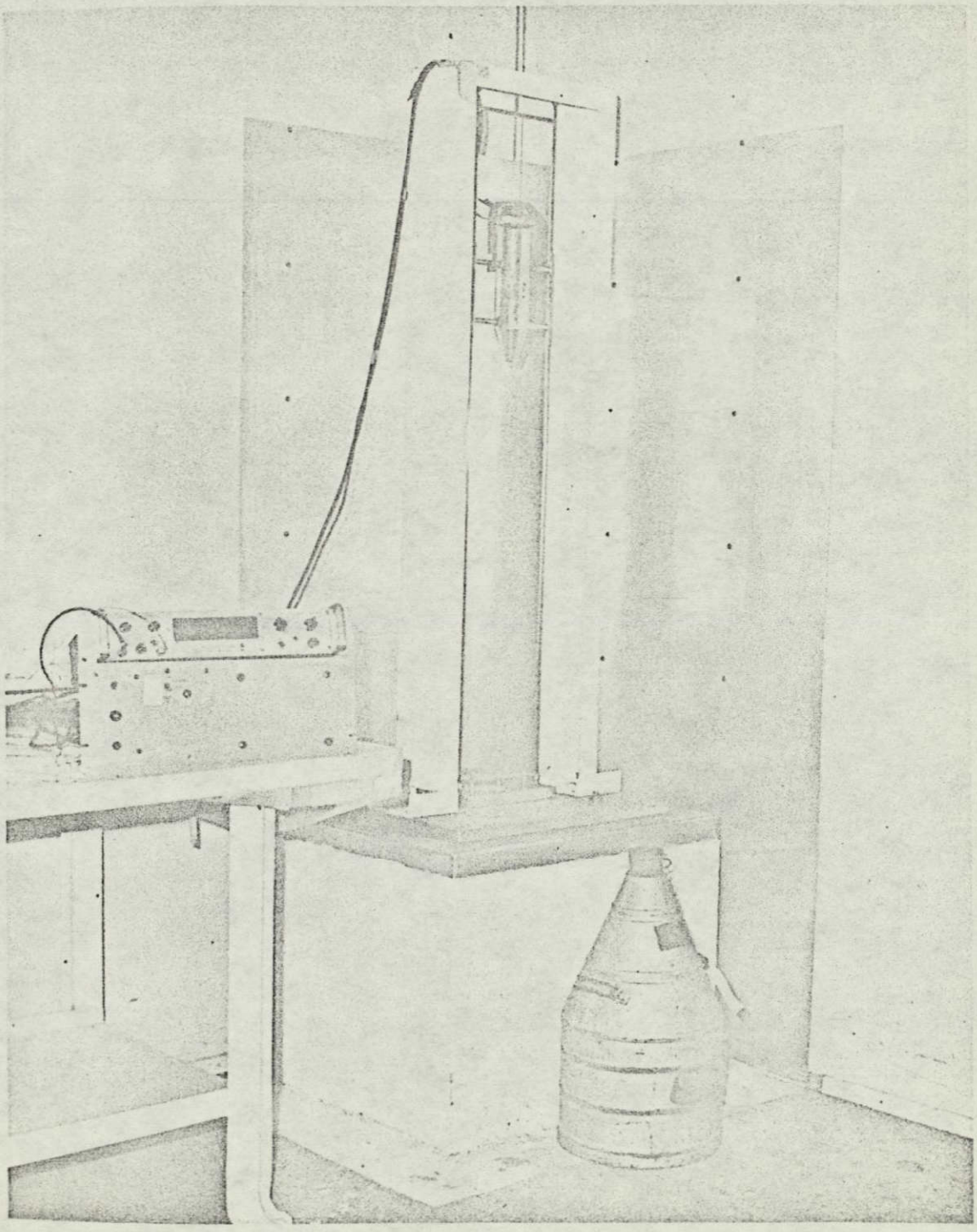

Figure 5-13. Liquid Oxygen ABMA Impact Test Setup

Table 5-6. ABMA LO_2 Impact Sensitivity Test Results, First Series

Material	Impact Load (ft-lb)					
	72	60	50	40	30	20
Teflon TFE (control)	0/10	-	-	-	-	-
255-2 Viton-A	3/3*	2/10*	0/4	0/2	-	-
AF-E-71-2 EPT Compound	3/3*	-	-	2/5	2/2*	1/1
263-2 EPT-Hydrin	3/3*	-	-	3/5	2/2	2/2
310-1 Phenyl Silicone	6/10	-	3/3	0/3	-	-
316-1 Fluorosilicone	3/3	-	2/4	0/3	-	-
1904-85 HYSTL	4/5	-	-	2/2	2/5	2/8

*Indicates an audible detonation.

Code: Fractions depict number of reactions/total impact tests.

As can be noted from the results of the first test series, all materials except Teflon flashed at the standard impact load of 72 ft-lb. As a consequence, the impact load was decreased to 40 ft-lb. At this level, the Viton-A and two silicone compounds were satisfactory with a low number of samples. The impact load was then increased in steps to 50 and 60 ft-lb until a reaction occurred. The two EPT compounds and the HYSTL were then tested at progressively lower loads of 30 and 20 ft-lb. Reactions occurred at each energy level on all three materials, although the HYSTL indicated less sensitivity. The second test series was intended to provide a larger sample quantity for the Viton at 50 ft-lb, the silicone compounds at 40 ft-lb and an additional 10 impacts at 72 ft-lb on the Teflon. As previously mentioned, this second test series was discontinued because of apparent impurities causing reactions.

The third series of impact sensitivity tests were run to supplement those previously conducted. These tests showed substantially the same results reported for the first test series. Tests were also conducted with the new AF-E-124D material which was the only material other than Teflon which showed no reaction at the 72 ft-lb impact level. This is equivalent to the Teflon TFE results, and supports the selection of this material as the most promising candidate for all-around use in cryogenic media. A summary of all the test results has been compiled and is presented in Table 5-7.

5.3 LIQUID HYDROGEN VALVE SEAL TESTS

Sealing capability at -423°F was verified by a series of tests which were conducted using a typical valve, essentially identical to that used for the liquid nitrogen tests (Section 5.1).

The test setup is shown schematically in Figure 5-14. These tests were conducted at Wyle Laboratories in Norco, California. A photo of the test setup in operation is shown in Figure 5-15. The insulated test valve and operator are shown, with a long stem cryogenic hand valve which was used to regulate the flow of liquid hydrogen through the test valve to maintain the liquid hydrogen temperature. In the foreground of this picture, a water bath heat exchanger, which was used to insure that the gas leakage measurement was at ambient temperature is shown.

Table 5-7. ABMA LO_2 Impact Sensitivity Test Results Summary

Material	Impact Load (ft-lb)						
	72	60	50	40	30	20	10
Teflon TFE (control)	0/40						
AF-E-124D	0/20						
255-2 Viton-A	3/3*	2/10*	8/46	0/2			
310-1 Phenyl Silicone	6/10	-	3/3	12/24	2/2*	2/20	
1904-85 HYSTL	4/5	-	-	2/2	2/5	2/8	1/20
316-1 Fluorosilicone	3/3	-	2/4	7/11*	3/3	4/10	1/10
AF-E-71-2 EPT-HYSTL	3/3*	-	-	2/5	2/2*	1/1	1/10
263-2 EPT-Hydrin	3/3*	-	-	3/5	2/2	2/2	2/10

*Indicates an audible detonation.

Code: Fractions depict number of reactions/total impact tests.

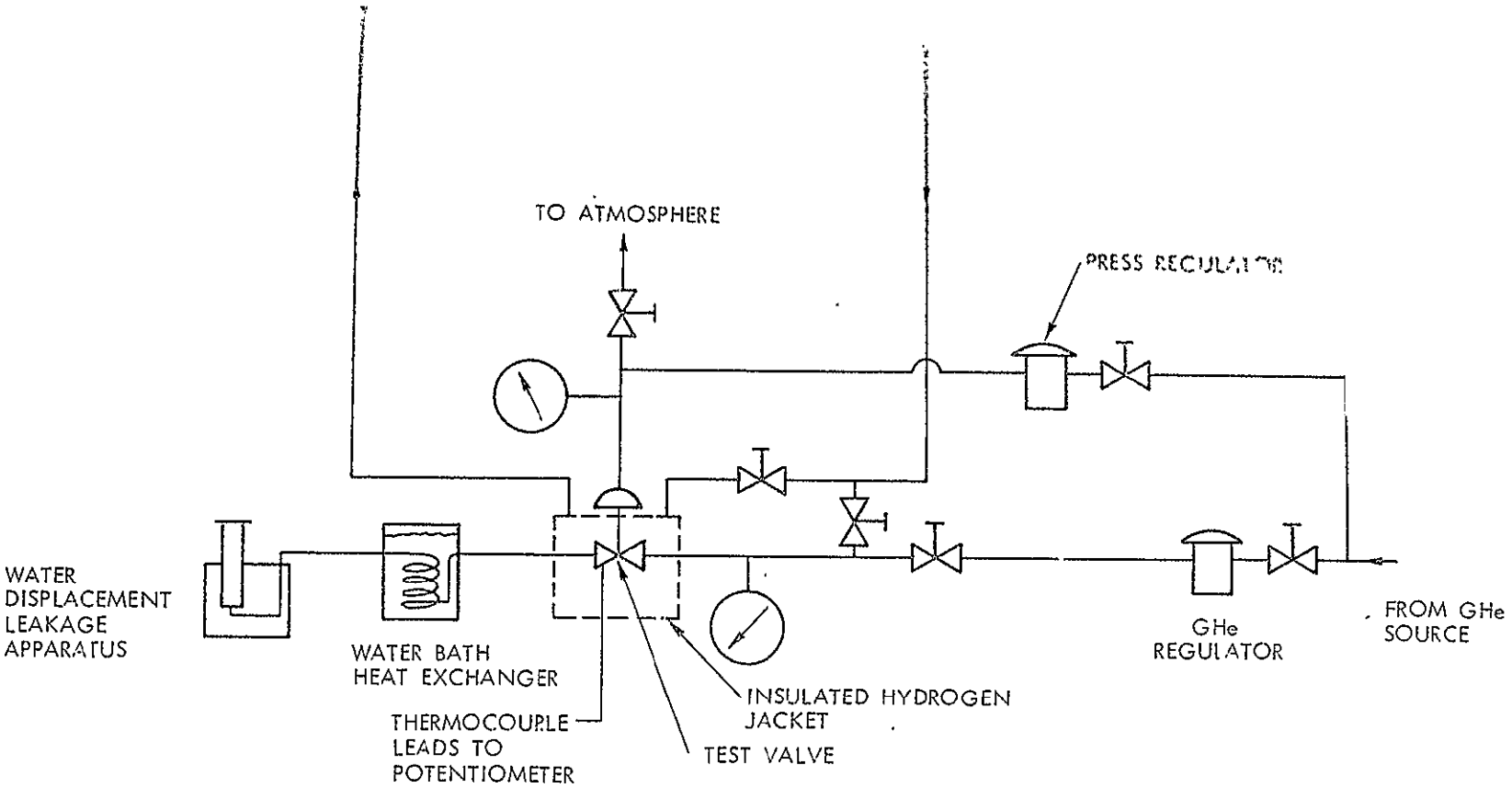

Figure 5-14. Liquid Hydrogen Test Schematic

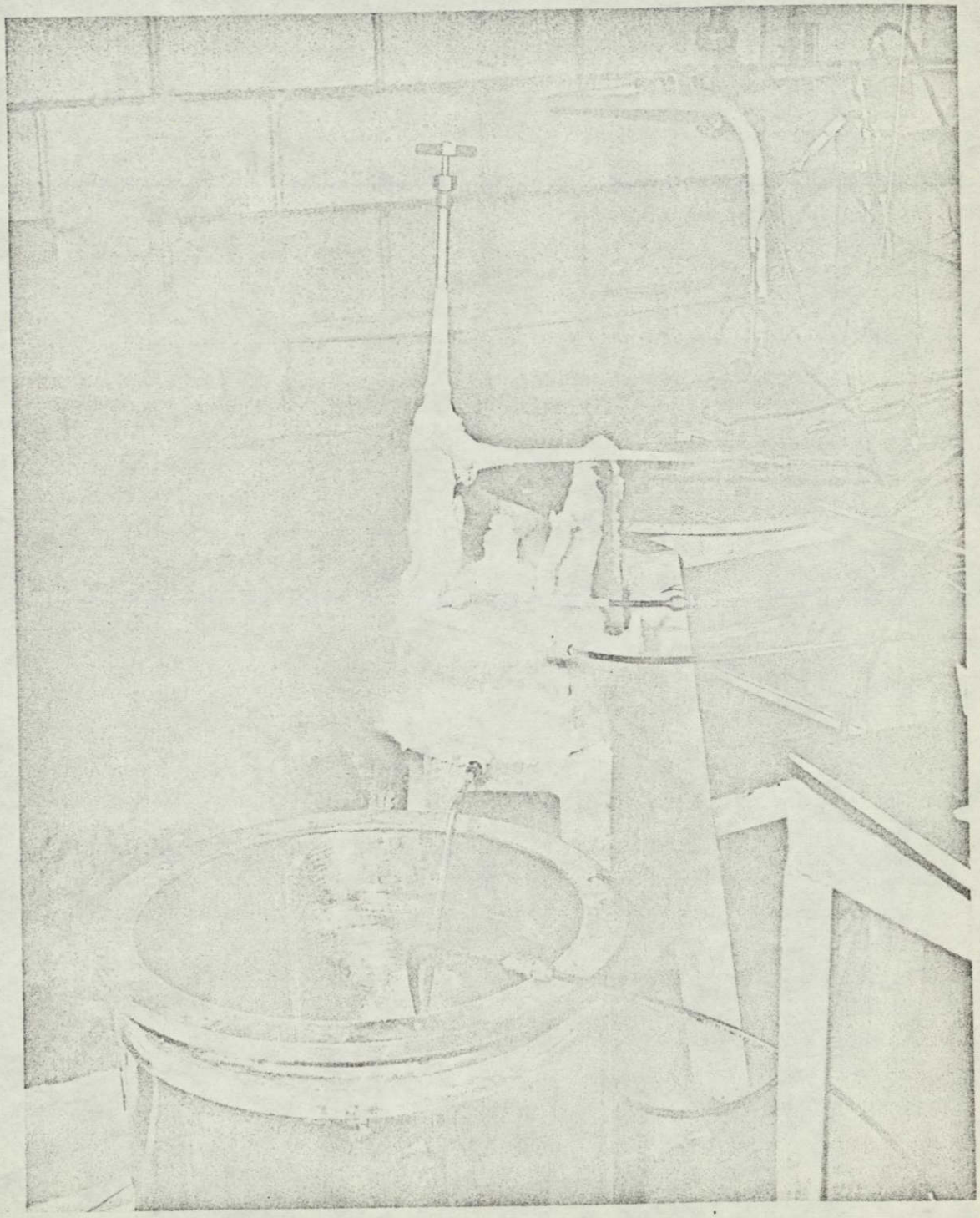

Figure 5-15. Liquid Hydrogen Seal Test Fixture

The general test setup is shown in Figure 5-16. As shown schematically in Figure 5-14, the liquid hydrogen is stored in a portable dewar shown to the left of Figure 5-16. The liquid hydrogen flows from the dewar to the test setup and is controlled by the manual cryogenic valve. The LH_2 then flows into a jacket around the test valve, which is filled completely when temperature stabilized with liquid hydrogen. To maintain liquid hydrogen temperature, the hydrogen flows through the jacket and out through a vent line to atmosphere. For the first series of tests, the test media was gaseous helium which was used to determine leakage through the valve at hydrogen temperatures. Figure 5-17 shows the test valve in the container which was closed and insulated making a jacket to retain LH_2 and cool the valve. Note that two thermocouples were welded to the bottom of the valve to indicate test valve temperature. Figure 5-18 provides an overall view of the controls used in testing, showing gauges used to measure inlet pressure, outlet pressure and hand loaders used to regulate the inlet pressure. Valve leakage was measured by standard water displacement methods. The line coming from the water bath heat exchanger was connected to a water displacement measurement apparatus which then measured the rate of internal leakage from the test valve.

The test procedure used in the first series of liquid hydrogen tests is as follows:

1) Determine the valve seat load (pressure on dome) required to effect zero leakage* with helium at ambient temperature at 200 psi and 400 psia inlet pressure.

2) Using helium, purge through the jacket and the valve for 15 minutes.

3) Flowing liquid hydrogen through the jacket, cool down to -423°F with a continuous helium purge through the test valve. (In some cases the temperature is held at some intermediate point to check LN_2 temperature results).

4) At -423°F determine the valve seat load (pressure on dome) to obtain zero leakage* through valve.

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*The term zero leakage, as used herein, denotes no discernable helium leakage for a period of 5 minutes using a water displacement measurement apparatus.

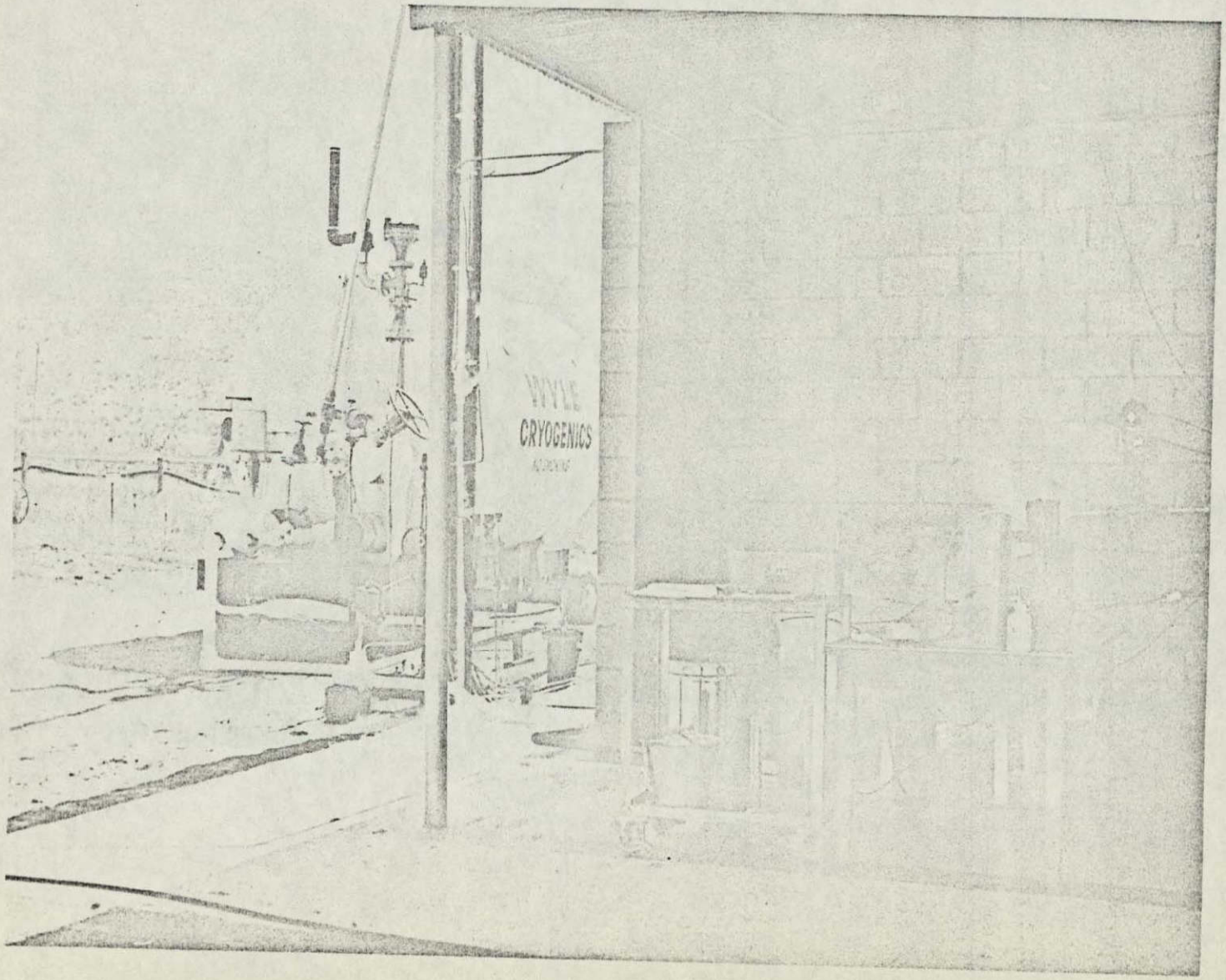

Figure 5-16. Liquid Hydrogen Seal Test Setup

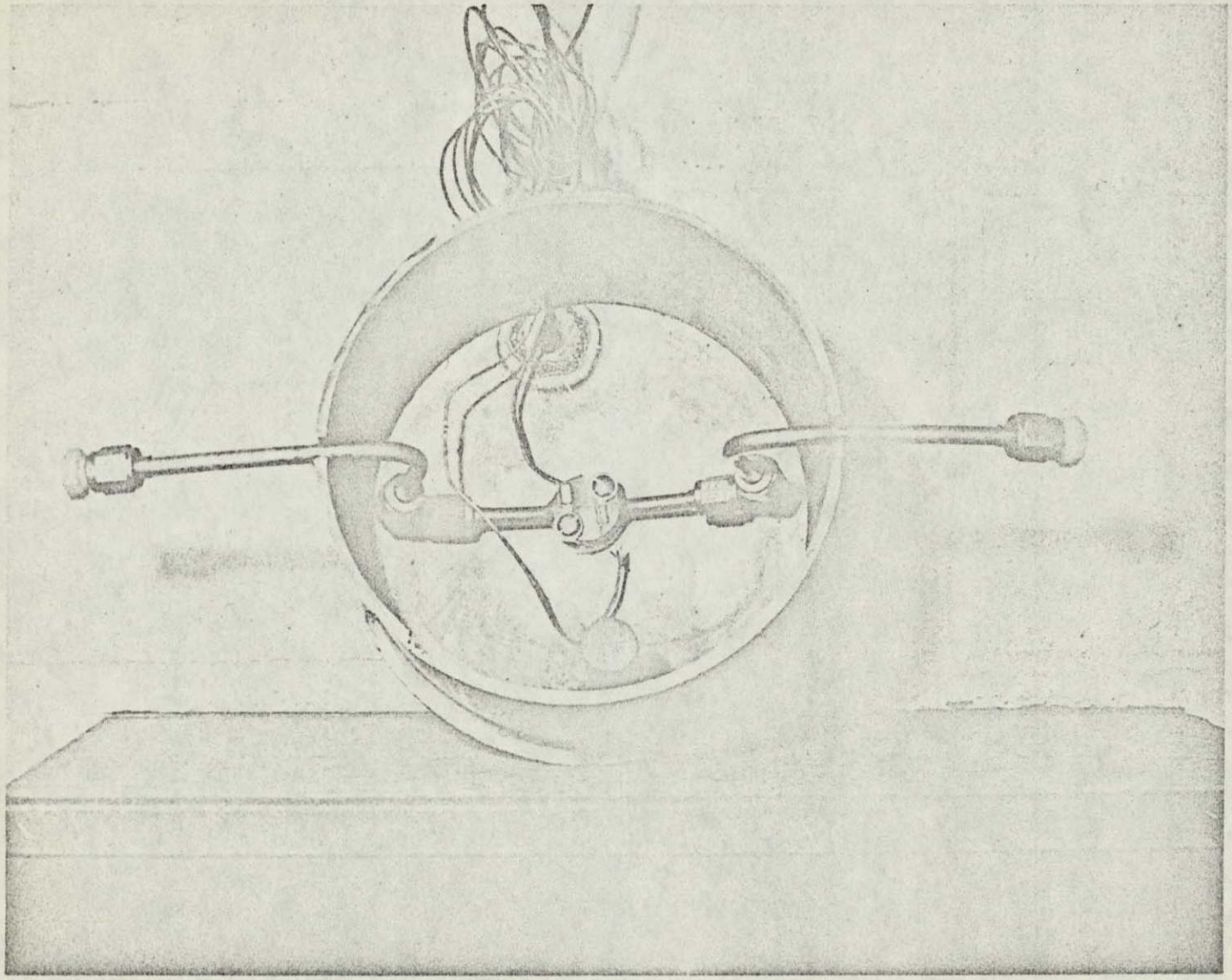

Figure 5-17. Liquid Hydrogen Test Valve and Jacket

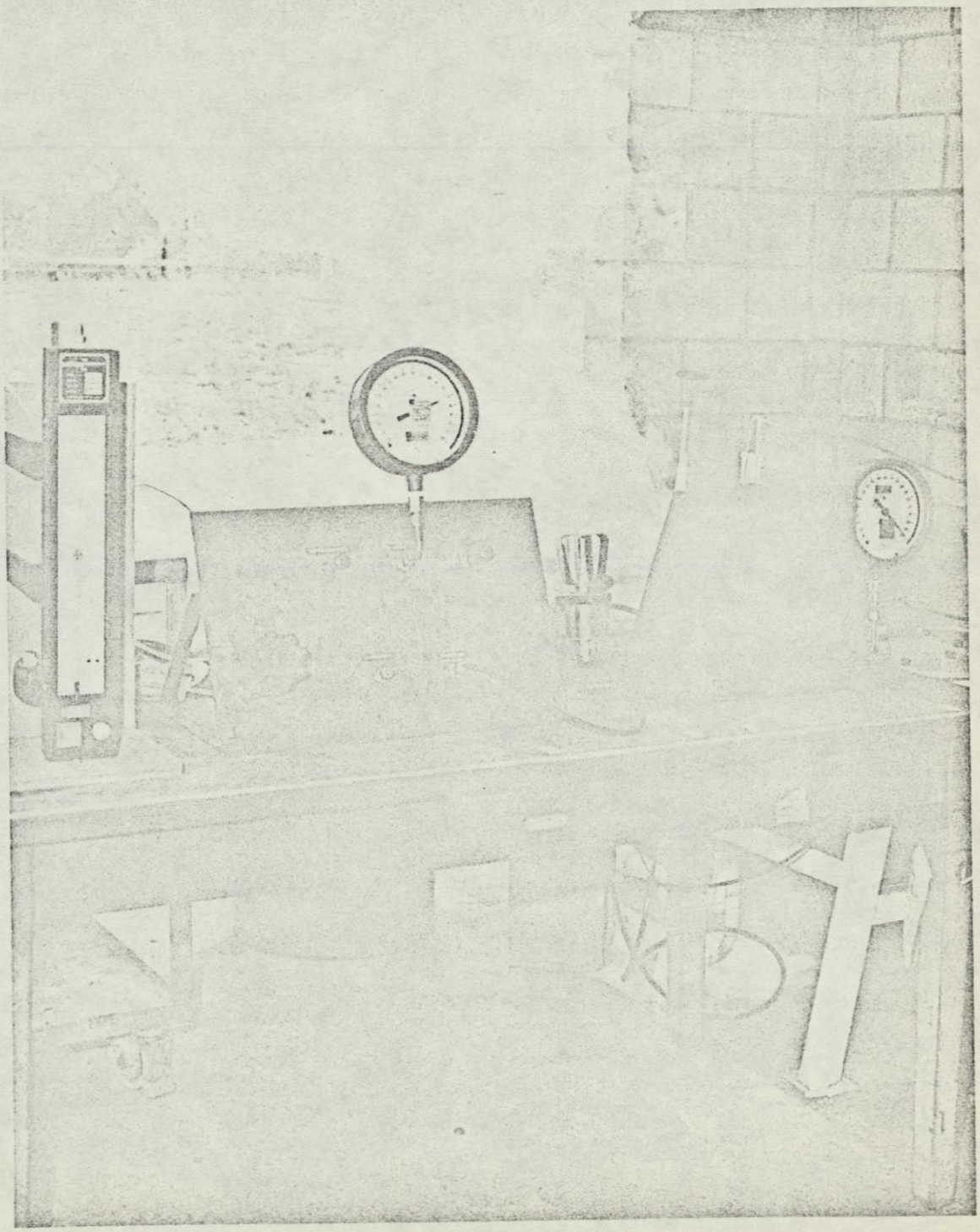

Figure 5-18. Liquid Hydrogen Test Pressurization Controls

5) Cycle valve (varies from zero to 100 cycles); again determine seat loads to effect zero leakage.*

6) Purge with helium and warm to approximately room ambient temperatures (ambient data were actually taken at temperatures from +35° to +70°F because of time limitations).

7) Obtain seat loads required to effect seal after warming to ambient temperatures.

The materials tested during this phase of the program were essentially those that had been subjected to the previous screening tests and appeared most applicable to the program goals. These materials were:

- Teflon TFE
- AF-E-124D
- Phenyl Silicone 310-1
- AF-E-71-2
- EPT/HYDRIN 263-2
- HYSTL
- Viton A
- Fluorosilicone 316-1

The composition, general description and attributes of these materials were discussed earlier in Section 4.

The final series of tests were conducted with liquid hydrogen flowing in the test valve (as opposed to the gaseous helium previously used) to determine if direct contact between the seat material and the liquid hydrogen would provide the same results as with helium. The test procedure was essentially the same with the exception that liquid hydrogen was flowed through the valve during the cool-down and test periods. The valve leakage rate, as determined by gaseous hydrogen leakage measured with the water

*The term zero leakage, as used herein, denotes no discernable helium leakage for a period of 5 minutes using a water displacement apparatus.

displacement method, was measured at cryogenic temperatures both before and after cycling. The valve was cycled 100 times to determine the change in seat load required to effect zero leakage in liquid hydrogen. Each of the materials previously tested was run at least once with liquid hydrogen with a number of materials being run two or three times.

The final step for all the liquid hydrogen tests was the visual inspection of the various seat materials after test to determine the relative cold flow or seat impact load characteristics as a result of the test series.

5.3.1 LH_2 Liquid Hydrogen Seat Test Results

The basic criteria for evaluation of materials subjected to LH_2 tests is:

1) Seat load required to effect a zero leakage seal

2 Visual appearance of material after being subject to exposure and cycling at LH_2 temperature

As a result of these tests, the materials are ranked as to their applicability for use as an LH_2 seal as derived from the above parameters.

The test valve was shown in Figure 5-15. A schematic of this valve was shown previously in Figure 5-12. The seal material in all cases is approximately 0.200 inch diameter and 0.100 inch thick. The ranking of these materials as to applicability for use as an LH_2 seal as determined from these tests is:

1) HYSTL

2) AF-E-124-D

3) Teflon TFE

4) Viton A 255-2

5) Phenyl Silicone 310-1

6) EPT/HYSTL AF-E-71-2

7) EPT/HYDRIN 263-2

8) Fluorosilicone 316-1

The test data from which the conclusions were reached are provided in Appendix A; a summation of the important results and material characteristics is shown in Table 5-8. It is important to note that much of the ranking is subjective and can only be considered as an approximate guide.

Fluorosilicone 316-1 is rated last because of consistent cracking at LH_2 temperature under static seat loads. In all the LH_2 tests, excessive leakage occurred at -423°F and could not be sealed with increased load. Post-test examination showed the seal cracked in each cas-

A summary of the seat loads and assessment of visual appearance afterward is given in Table 5-8.

Table 5-8. Summary of LH_2 Test Results

Material	Seat Stress to Effect Seal		Post-Test Condition
	Ambient (psi	-423°F (psi)	---
HYSTL	300	1270	No visual seat impression
AF-E-124D	600	4200	No visual seat impression
Viton A (255-2)	300	3050	Very light seat impression
Teflon TFE	600	6900	Deep seat impression
Phenyl Silicone (310-1)	1000	4300	Light seat impression
AF-E-71-2	300	3470	Medium seat impression (one sample partially) cracked)
EPT/HYDRIN (263-2)	460	3000	Deep seat imprint
Fluorosilicone (316-1)	300	*	Material cracked

*No seal effected.

Although it was difficult to obtain realistic seat impression photographs, Figures 5-19 through 5-25 give a relative comparison of the seat conditions after being subjected to equal test conditions.

The data provided in Table 5-8 reflects average data derived from various test samples. The apparent significant superiority of the HYSTL over the other materials in the group was unexpected and may not be a result of relative materials capability. Since HYSTL is a plastic, the surface finish is normally very smooth. At the other extreme, the AF-E-124D samples available for test (since material is in limited supply) had relatively uneven surfaces, as may be seen in Figure 5-19. This may account for the variation in seat stress required to effect a seal as shown in Table 5-8 both at ambient temperature and -423^{O}F. Other material surfaces were of approximately equal quality with the HYSTL surface.

5.4 SOLVENT AND CLEANING FLUID EXPOSURE TEST DESCRIPTION

Each candidate seal material was subjected to a series of solvent exposure tests to establish fluids compatibility. The fluids used in this test are common cleaning and test fluids normally used in propellant systems. They are:

- Distilled water
- Freon TF
- Isopropyl Alcohol (IPA)
- Trichlorethylene

The test procedures used were as follows: First, samples were obtained in standard test sample sizes. Measurements of length, thickness and weight were taken. The samples were placed in individual containers with each fluid. The samples were exposed to the fluids for the following lengths of time:

- One hour
- Four hours
- One day
- Three days
- Seven days
- Fourteen days

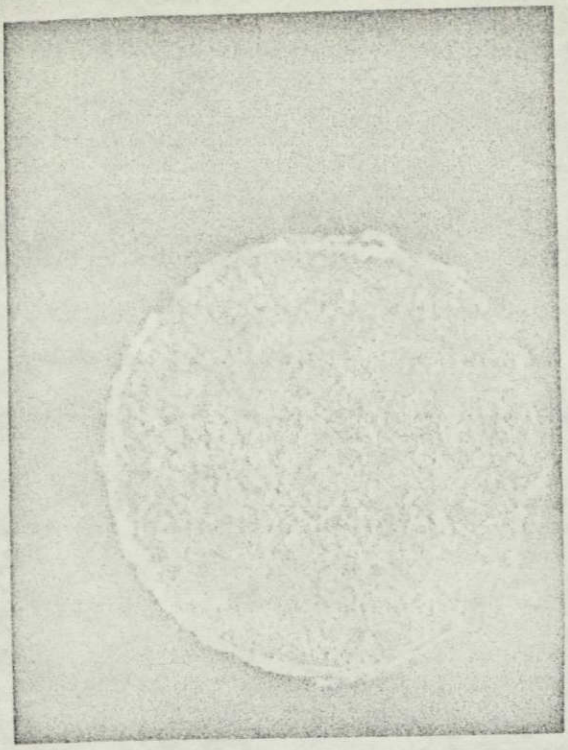

Figure 5-19. Experimental Fluoroelastomer AF-E-124D

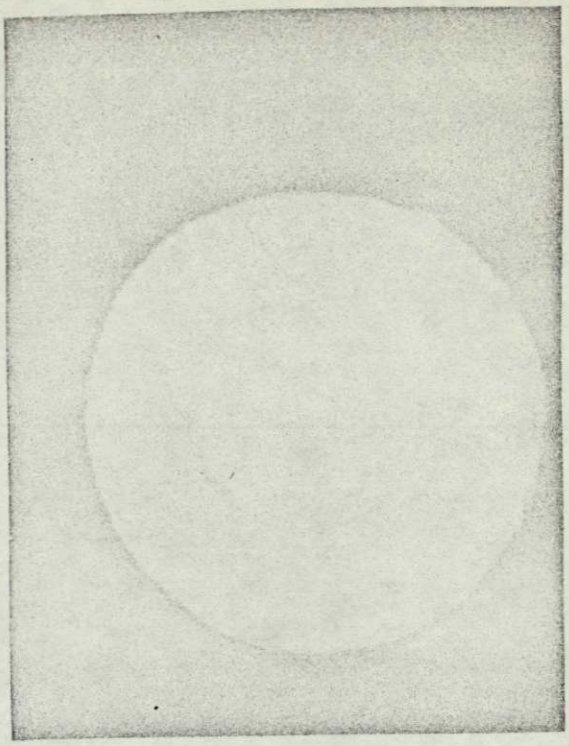

Figure 5-20. Teflon TFE

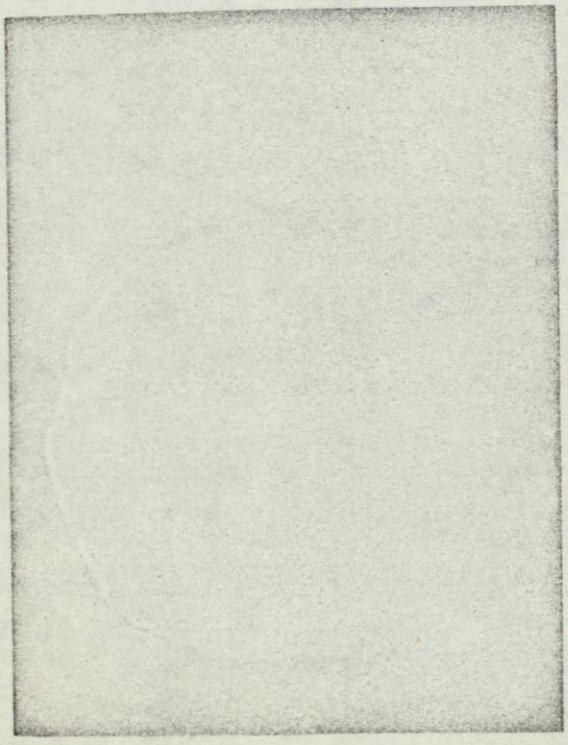

Figure 5-21. Viton A

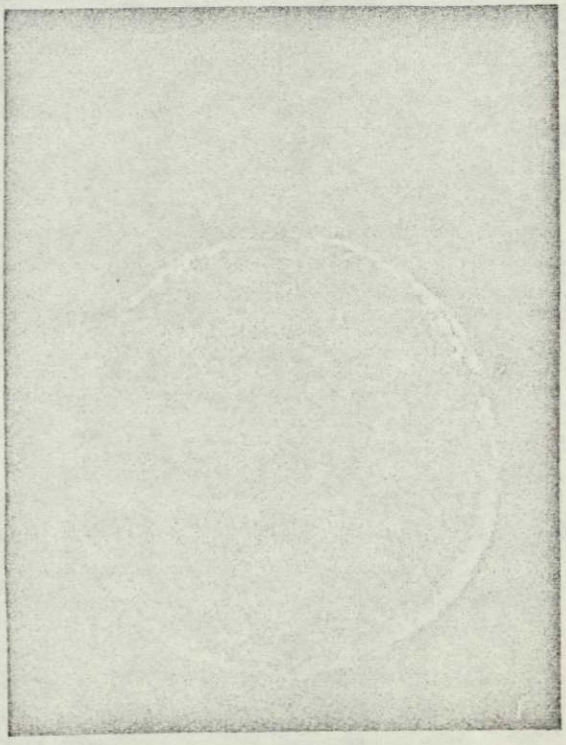

Figure 5-22. HYSTL

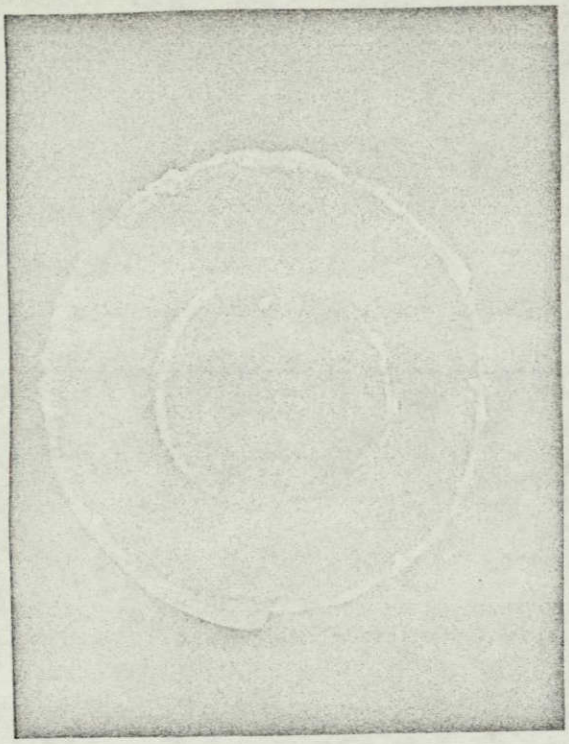

Figure 5-23. AF-E-71-2

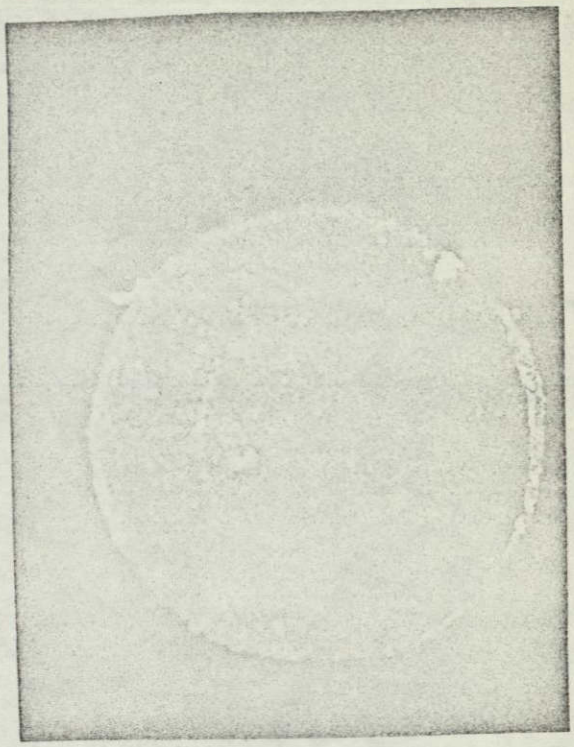

Figure 5-24. Phenyl Silicone 310-1

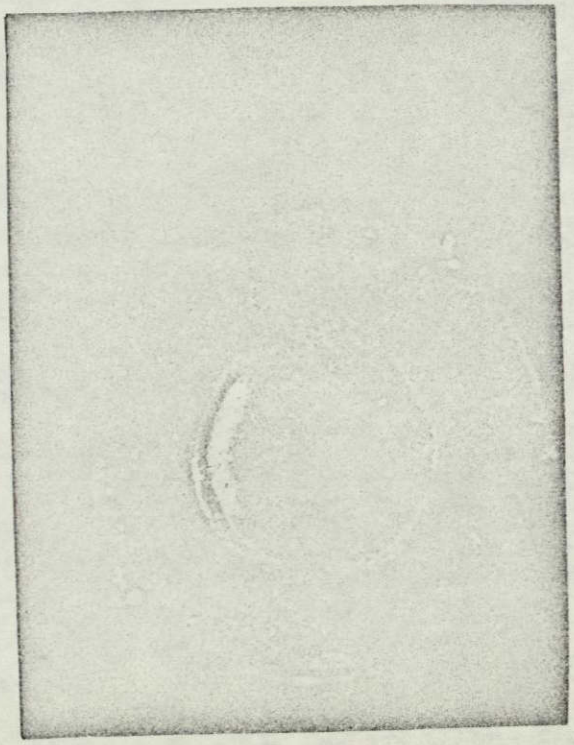

Figure 5-25. EPT/HYDRIN 263-2

At the end of each exposure period the samples were removed from the solvent, wiped dry and immediately weighed and remeasured. The samples were then allowed to air dry for approximately one week, at the end of which time they were again reweighed and remeasured. The final step in this series was to subject each sample to a tensile test to determine the ultimate strength and the elongation of the sample at rupture, as a measure of the mechanical property changes as a result of solvent exposure.

5.4.1 Solvent and Cleaning Fluid Test Results

As anticipated the results of this test series indicated that not all the materials are compatible with common presently used cleaning solvents and flushing fluids. A general summary is provided in Table 5-9 indicating recommended (R) or not recommended (NR) for each material and solvent. This is a conservative evaluation since time of exposure is also a factor and if incompatibility was indicated during the longest exposure (14 days) then an NR is listed. The data in Appendix C provide more detailed information on time and degree of incompatibility. In general, of the materials which have application in both O_2 and H_2 systems, Teflon is compatible with all the fluids, AF-E-124 indicates some incompatibility with Freon TF and trichlorethylene, and Viton A is compatible with all fluids.

5.5 ELEVATED TEMPERATURE EXPOSURE TESTS

The elevated temperature exposure tests were conducted using the same condidate materials as for the solvent exposure tests. The temperature tests consisted of heating the specimens in an air oven for exposure times of 16 hours, 3 days, 10 days and 21 days to +250°F, +325°F, and +400°F.

The length, thickness, and weight of each specimen was established prior to test and immediately after test for each exposure period. Each sample was then subjected to a tensile test similar to the solvent exposure series to determine the ultimate strength and elongation at rupture to determine any degradation of mechanical properties as a result of exposure to high temperature.

Table 5-9. Summary of Cleaning Solvents and Flushing Fluids

Material	Distilled Water	Freon TF	Isopropyl Alcohol	Trichlorethylene
AF-E-124D	R	NR	R	NR
Teflon TFE	R	R	R	R
Viton A (255-2)	R	R	R	R
HYSTL	R	R	R	NR
Phenyl Silicone (310-1)	R	NR	R	NR
AF-E-71-2	R**	R	R*	NR
EPT/HYDRIN (263-2)	R	R	R	NR
Fluorosilicone (316-1)	R	NR	R	NR

R - Recommended

NR - Not Recommended

* - Some degradation of physical properties observed

** - Short term only degradation noted.

5.5.1 Elevated Temperature Test Results

The purpose of these tests was to determine material degradation after exposure to the elevated temperature as a measure of capability at temperature. These can be interpreted as screening tests for materials but not necessarily verification of material capability at temperature. An example of this is Teflon. From the summary provided in Table 5-10, the Teflon indicates no change in tensile strength after exposure to 400°F. However, the general characteristics of Teflon are such that a high degree of deformation under load would occur at this temperature, even in a well contained seal design. The results shown in Table 5-10 are a summary of room temperature property changes after exposure to temperature for 10 days. In Appendix C actual values are shown as well as properties after exposure for 16 hours, 3 days and 21 days. HYSTL properties were

established by using flexural properties rather than tensile tests. This was necessary because of the specimen shape and the brittle facture characteristic of HYSTL where elongation has no meaning. The values used are: Modulus of Rupture ($S_v = \frac{Mc}{I}$) and modulus of elasticity.

Table 5-10. Elevated Temperature Exposure Test Results (after 10 days exposure - change in room temperature properties after exposure)

Material	250°F		325°F		400°F	
	Elongation (% Change)	Ultimate (% Change)	Elongation (%)	Ultimate (%)	Elongation (%)	Ultimate (%)
AF-E-124D	+45	-27	+32	-30	+48	-20
Teflon TFE	+22	0	+10	0	+8	+4
Viton A (255-2)	0	-15	-12	0	-14	0
Phenylsilicone (310-1)	-8	-200	-14	-11	-50	-32
HYSTL	+13	0	-5	+16	-13	0
Fluorosilicone (316-1)	-10	-25	-19	-30	-42	-42
AF-E-71-2 (EPT)	-75	-54	-90	-80	-90	-93
EPT/HYDRIN (263-2)	-31	+20	*	*	*	*

*Specimens Physically Decomposed

6. SEAL DESIGN CONSIDERATIONS

6.1 SEALING LOAD

The seal load is the total force applied normal to the seal interfaces expressed in pounds. Two major factors which influence the mating at the seal interfaces are (1) the initial topography of the mating surfaces, and (2) the interface deformation resulting from seal load. In general, the better the finish of the mating surfaces, the lower the stress (seal load per unit area) required for achieving a seal.

Elastomers, unlike metals, deform by large elastic deformations to mate with rigid surfaces, and hence are less critical with respect to surface finishes. The seal stress required to achieve surface mating is extremely small. Often seal stresses as low as 500 psi are adequate to effect a seal. Since the deformation of the seal is almost entirely elastic, the initially applied seating load must be maintained. Thus, an additional load margin must be applied to allow for strain relaxation during the life of the seal.

Plastics, like elastomers are readily deformed against hard metal surfaces thereby minimizing the importance of good surfaces for static sealing. For dynamic sealing, surface finishes become important for reasons other than sealing, namely friction and wear. The deformation in plastics is of viscoelastic nature; thus, without proper constraint, the phenomenon of cold flow can exist, resulting in unsatisfactory design caused by sealing relaxation. Preventing of bulk flow of the plastic material requires adequate containment of the seal by the gland. In general, minimum seating stress levels of approximately 0.7 times the yield strength of the plastic will be sufficient to effect a seal.

6.2 ELASTICITY

A seal must be designed with the capability of storing elastic energy to allow the seal to maintain interface loads under conditions of gland deformation. If the seal material itself is not inherently elastic, the overall seal design must include a springlike member.

6.3 STRENGTH

The seal must be capable of resisting the differential pressure across it, to eliminate the possibility of blowout or wire drawing. Design techniques such as the use of backup rings can be used to effectively increase the strength of some seal materials.

6.4 FRICTION AND WEAR

In dynamic seals, abrasion and tearing of the interface materials are primary reasons for seal failure. Characteristics which influence the rate of wear include surface finish and hardness; and functional characteristics at the interface. The amount of time the seal is at rest and functioning as a static seal will have a significant effect on the degree of mating and level of friction loads. Friction load may be decreased by decreasing unit load, differential pressure, surface finish, as well as by the use of lubrication.

7. CONCLUSIONS AND RECOMMENDATIONS

The fluoroelastomer AF-E-124D emerged from this program as a significantly superior material for cryogenic seal applications. In all the evaluations conducted, this material was generally equal or superior to Teflon TFE which is generally considered the state of the art material for cryogenic service. No significant deficiencies or degradation was noted in all the tests conducted except for some cleaning fluids. Table 7-1 is a summary of the performance of AF-E-124D in all the tests as compared with Teflon. Note that the property data are approximately equal with the exception that Teflon is compatible with more cleaning fluids, while the AF-E-124D exhibits no observable compression set in valve tests at ambient and no degradation during low temperature testing. A significant advantage of AF-E-124D over Teflon is the elevated temperature load deformation characteristics. Teflon exhibits appreciable deformation at 200°F under load, while limited data on AF-E-124D indicates no significant change.

Table 7-2 is a summary of the materials (presented earlier in Section 1, Summary) in order of applicability for an overall seal material and the relative performance of each in various test series.

Recommendations for the continuing effort on this program fall into the following categories:

1) Continuation of characterization of the AF-E-124D base stock material; and compounding additional materials for evaluation.

2) Incorporation of AF-E-124D, Viton A, Hystl and Teflon TFE in various valve and static seal designs for evaluation of "in-service" conditions with LH_2 and/or LO_2.

3) Determination of high temperature oxygen compatibility and impact sensitivity.

4) Evaluation of the effects of cleaning solvent residue on LO_2 impact sensitivity.

Table 7-1. Comparison of AF-E-124D and Teflon TFE

	AF-E-124D		Teflon TFE	
	Initial or Ambient	Post Test	Initial or Ambient	Post Tes
1. Tensile Strength after Exposure to:				
250°F	2500 psi	1812	2930 psi	2915
325°F	2500 psi	1730	2930 psi	3080
400°F	2500 psi	2000	2930 psi	2900
Distilled Water	2500 psi		2930 psi	2800
Freon TF	2500 psi	(1)	2930 psi	2930
Trichlorethylene	2500 psi		2930 psi	3100
Isopropyl Alcohol	2500 psi		2930 psi	2890
2. Seat Stress to Seal				
LH_2 Temperature	630	5200 2320	578 psi	6900 psi
LN_2 Temperature	630	3750	578 psi	2140 psi
3. Compression Set after LH_2 Valve Seal Tests	None	None	None (initial)	Extreme
4. Hardness	78 Shore A	(1)	Rockwell 58R	(1)
5. Modulus of Elasticity	835	(1)	580,000	(1)

(1) Not Determined

Table 7-2. Summary of Seal Material Performance

Rating			Material	Sealing Capability Ratio of Load Required to Seal at Cryogenic Temperature to Ambient Temperature		LO_2 Impact Resistance Maximum Impact with no Reaction ft-lbs	Resistance to Permanent Seat Deformation (3)	High Temperature Resistance, Average Percent Change in Tensile Strength	
H_2	O_2	O_2/H_2		LH_2	LN_2				
							+60 to -423°F	+400°F	+250°F
2	1	1	AF-E-124D	6.0	6.5	72	Excellent	-20	-27
4	2	2	Teflon TFE	10.0	3.7	72	Fair	0	0
3	6	6	255-2 (Viton A)	5.0	4.5	40	Good	0	-12
8	7	7	310-1 (Phenyl Silicone)	4.7	3.4	<20	Good	-26	-14
1	***	***	HYSTL	3.7	2.9	<10	Excellent	0	0
**	***	***	316-1 (Fluoro-silicone)	**	12.5	<10	Poor	-50	-28
12	***	***	AF-E-71-2	11.6	6.0	<10	Fair	-93	-54
11	***	***	263-2 (EPT-HYDRIN)	6.3	(1)	<10	Fair	*	0
9	***	***	Mylar (2)						
5	3	3	Kel-F (2)						
16	***	***	Nylon (2)						
7	5	5	Kynar (2)						
6	4	4	Vespel (2)						

*Material Decomposed.

**Material failed under load.

***Not rated due to LO_2 impact sensitivity

(1) Not tested.

(2) Not tested during this program

(3) Definition of terms:
Excellent - No evidence of permanent set
Good - Slight seat imprint
Fair - Medium to deep seat imprint
Poor - Seat cracked

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APPENDIX A

LIQUID HYDROGEN

TEST DATA

LH_2 Seal Test Data

Material	Temperature (°F)	Inlet Pressure (psi)	Valve Dome Load to Effect Zero Leakage (lbs)	Seal Stress (psi)
Teflon TFE	+70	400	10	578
	-320	400	37	2140
	-423 (GHe)	400	120	6900
	-423 (LH_2)	200	80	4620
AF-E-124D	+70	400	11	630
	-320	400	65	3750
	-423 (GHe)	400	40	2320
	-423 (LH_2)	200	90	5200
Viton A 255-2	+70	400	1.0	60
	-295	400	47	2720
	-423 (GHe)	400	53	3050
	-423 (LH_2)	200	-	-
HYSTL	+70	400	6	345
	-320	400	18	1020
	-423 (GHe)	400	21	1270
	-423 (LH_2)	200	20	1150
AF-E-71-2	+32	400	5	290
	-320	400	30	1735
	-423 (GHe)	400	60	3470
	-423 (LH_2)	200	-	-
Phenyl Silicone 310-1	+47	400	16	925
	-320	400	54	3120
	-423 (GHe)	400	75	4340
	-423 (LH_2)	200	100	5780
Fluorosilicone 316-1	+55	400	8	460
	-320	400	100	5780
	-423 (GHe)	400	-	Material Cracked
	-423 (LH_2)	200	-	Not Tested
EPT-Hydrin 263-2	+65	400	8	460
	-320	400	-	-
	-423 (GHe)	400	50	2900
	-423 (LH_2)	200	-	-

APPENDIX B

LN_2 COMPRESSION LOAD TEST DATA

Sample Description	Compression (Inches)	Load Pounds	Temperature °F
255-2	0.0200	94.7	70
Viton-A	0.0272	127.0	70
	0.0358	18.3*	-320
Initial Thickness: 0.236	0.0314	72.1*	70
Final Thickness: 0.233	0.0300	47.0	70
Diameter: 0.863	0.0278	30.5	70
Area: 0.585	0.0256	16.5	70
(11.2 percent Compression)	0.0100	0	70
255-2	0.0152	55.0	70
Viton-A	0.0245	95.3	70
	0.0359	140.5	70
Initial Thickness: 0.235	0.0457	198.5	70
Final Thickness: 0.229	0.0625	284.0	70
Diameter: 0.861	0.0625	175.3*	70
Area: 0.582	0.0631	67.2*	-320
	0.0634	177.1*	70
	0.0623	103.8	70
(26.7 percent Compression)	0.0600	69.0	70
	0.0570	37.9	70
	0.0541	25.6	70
	0.0447	12.2	70
	0.0263	0	70
263-3	0.0084	55.0	70
EPT-HYDRIN	0.0113	85.5	70
	0.0135	128.2	70
Initial Thickness: 0.237	0.0214	216.8	70
Final Thickness: 0.234	0.0286	282.8	70
Diameter: 0.862	0.0330	219.9*	70
Area: 0.584	0.0341	14.7*	-320
	0.0314	224.7*	70
	0.0284	113.6	70
(12.1 percent Compression)	0.0277	83.7	70
	0.0224	33.0	70
	0.0195	15.3	70
	0.0133	0	70

*Load Allowed to Stabilize

Sample Description	Compression (Inches)	Load Pounds	Temperature °F
263-3	0.0093	78.2	70
EPT-HYDRIN	0.0170	174.0	70
	0.0279	269.9	70
Initial Thickness: 0.237	0.0364	360.3	70
Final Thickness: 0.228	0.0474	445.2	70
Diameter: 0.861	0.0533	519.1	70
Area: 0.582	0.0599	577.0	70
	0.0640	461.7*	70
	0.0672	128.2*	-320
	0.0644	439.7*	70
(25.3 percent Compression)	0.0599	310.8	70
	0.0583	241.8	70
	0.0551	147.2	70
	0.0511	102.6	70
	0.0429	46.4	70
	0.0328	15.3	70
	0.0166	0	70
	0.0134	95.3	70
AF-E-71-2	0.0228	182.0	70
EPT-HYSTL	0.0363	280.9	70
	0.0463	368.9	70
Initial Thickness: 0.232	0.0586	475.7	70
Final Thickness: 0.228	0.0586	375.6*	70
Diameter: 0.850	0.0586	102.0*	-320
Area: 0.567	0.0586	360.3*	70
	0.0569	240.6	70
	0.0543	158.2	70
(25.2 percent Compression)	0.0493	101.4	70
	0.0453	66.6	70
	0.0353	26.3	70
	0.0280	15.3	70
	0.0158	0	70
AF-E-71-2	0.0155	73.3	70
EPT-HYSTL	0.0260	139.2	70
	0.0408	207.6	70
Initial Thickness: 0.495	0.0546	291.3	70
Final Thickness: 0.488	0.0708	357.9	70
Diameter: 1.111	0.0855	435.4	70
Area: 0.950	0.0998	536.8	70
	0.1097	591.8	70
(22.2 percent Compression)	0.1119	439.7*	70
	0.1142	141.7**	-320
	0.1133	421.4*	70
	0.1040	266.9	70
	0.0986	176.5	70

*Load allowed to stabilize.

**Stabilization was not achieved after 1.7 hours.

Sample Description	Compression (Inches)	Load Pounds	Temperature °F
	0.0917	114.8	70
	0.0821	91.6	70
	0.0831	64.1	70
	0.0605	31.1	70
	0.0242	0	70
60-7	0.0134	73.3	70
Polybutadiene	0.0243	122.1	70
	0.0325	152.7*	70
Initial Thickness: 0.248	0.0349	116.0*	70
Final Thickness: 0.244	0.0334	6.1*	-320
Diameter: 0.860	0.0325	99.5*	70
Area: 0.581	0.0237	26.3	70
	0.0173	6.7	70
(13.1 percent Compression)	0.0091	0	70
60-7			
Polybutadiene	0.0093	55.0	70
	0.0240	134.4	70
Initial Thickness: 0.244	0.0406	183.2	70
Final Thickness: 0.241	0.0506	216.2	70
Diameter: 0.862	0.0644	268.7	70
Area: 0.584	0.0673	207.6*	70
	0.0709	6.1*	-320
	0.0721	180.2*	70
(29 percent Compression)	0.0657	119.1	70
	0.0579	76.9	70
	0.0517	36.6	70
	0.0442	22.0	70
	0.0174	0	70
215-2	0.0099	61.1	70
Buna-N	0.0222	102.0	70
	0.0288	103.8*	70
Initial Thickness: 0.238	0.0299	46.4*	70
Final Thickness: 0.234	0.0277	6.1*	-320
Diameter: 0.857	0.0305	43.4*	70
Area: 0.577	0.0264	15.3	70
	0.0254	8.5	70
(12.1 percent Compression)	0.0177	0	70

*Load allowed to stabilize.

Sample Description	Compression (Inches)	Load Pounds	Temperature °F
Teflon-TFE	0.0071	59.8	70
MIL-P-19468	0.0111	201.5	70
	0.0162	418.3	70
Initial Thickness: 0.243	0.0206	548.4	70
Final Thickness: 0.235	0.0213	605.8*	70
Diameter: 0.843	0.0228	465.4*	70
Area: 0.558	0.0175	152.7*	-320
	0.0210	406.1*	70
	0.0219	293.1	70
(9 percent Compression)	0.0210	175.3	70
	0.0199	106.9	70
	0.0193	57.4	70
	0.0182	18.3	70
	0.0115	0	70
310-1	0.057	268*	58
Phenyl Silicone	0.057	226*	58
	0.060	208	23
1/4 inch Thick	0.060	190	4
7/8 inch Diameter	0.060	190	- 25
	0.060	183	- 50
(24 percent Compression)	0.060	183	- 76
	0.061	178	- 97
	0.062	178	-114
	0.062	110	-167
	0.061	128	-220
	0.061	128	-262
	0.061	128	-237
	0.062	128	-222
	0.062	128	-180
	0.062	128	-167
	0.060	128	-143
	0.058	128	-125
	0.057	134	-108
	0.056	134	- 87
	0.056	134	- 65
	0.054	134	- 45
	0.054	128	- 25
	0.056	134	45
After 24 Hours	0.058	134	60

*Load allowed to stabilize.

APPENDIX C

SOLVENT CLEANING

FLUID COMPATIBILITY AND ELEVATED

TEMPERATURE TEST DATA

DIMENSIONAL CHANGE RESULTING FROM SOLVENT EXPOSURE

Material eflon TFE	Δ Weight Change Init x 10^{-4} gr	1 Week x 10^{-4} gr	Δ Length Change Init x 10^{-3} in.	1 Week x 10^{-3} in.	Δ Thick. Change Init x 10^{-3} in.	1 Week x 10^{-3} in.
Distilled Water						
1 hr	+3	0	-2	0	0	0
4 hrs	+3	+2	-1	-1	-1	-1
24 hrs	0	0	-3	-3	-1	-1
3 days	+2	+7	-11	+2	+2	+1
7 days	0	-2	-7	-11	+1	+1
14 days	+3	+3	-6	-8	0	0
Freon TF						
1 hr	+10	-6	-7	-5	0	0
4 hrs	+33	+6	-1	-1	-1	-1
24 hrs	+75	+39	0	-3	0	+1
3 days	+124	+78	-1	-1	+1	+3
7 days	+200	+113	-1	-2	0	0
14 days	+272	+180	-7	-9	0	0
Isopropyl Alcohol						
1 hr	-1	-4	-2	+1	0	0
4 hr	-2	-1	0	-3	0	0
24 hrs	-1	+1	-6	-1	-1	0
3 days	-2	-10	0	-3	+1	+1
7 days	0	-3	-4	-7	0	0
14	0	0	-8	-9	-1	-1
Trichlorethylene						
1 hr	-2	-4	-9	-9	0	0
4 hr	+11	-2	-3	-4	+1	+1
24 hrs	+24	+11	-10	-8	0	0
3 days	+32	+1	-6	-8	0	0
7 days	+62	+15	-3	-3	+2	+2
14 days	+67	+32	-1	-3	0	+2

NOTE: 1. "Initial" column indicates condition as removed from media.
2. "One Week" data indicates condition after 1 week air dry.

DIMENSIONAL CHANGES RESULTING FROM SOLVENT EXPOSURE

Material Viton A	Δ Wt		Δ Length		Δ Thickness	
	Init	1 Week	Init	1 Week	Init	1 Week
	x 10^{-4} gr	x 10^{-4} gr	x 10^{-3} in.	x 10^{-3} in.	x 10^{-3} in.	x 10^{-3} in.
Distilled Water						
1 hr	+4	+2	+3	+5	0	0
4 hrs	+3	+10	-1	+1	0	0
24 hrs	+26	+27	0	-1	0	0
3 days	+49	+40	+3	+4	0	0
7 days	+104	+68	0	0	0	0
14 days	+169	+115	+6	+4	0	+1
Freon TF						
1 hr	+86	+9	-4	-2	+1	+1
4 hrs	+818	+64	+1	-1	+1	0
24 hrs	+452	+194	+10	+6	+3	+2
3 days	+214	+412	+23	+15	+2	0
7 days	+991	+506	+42	+19	+4	+2
14 days	+1420	+921	+59	+34	+5	+4
Isopropyl Alcohol						
1 hr	+3	+15	-3	-3	0	0
4 hrs	+6	+17	-3	-1	0	0
24 hrs	+12	+2	+4	+3	0	0
3 days	+32	+35	-10	-10	-9	-9
7 days	+50	+178	0	0	0	0
14 days	+33	+37	+7	+4	+1	+1
Trichlorethylene						
1 hr	+49	+10	-5	-7	0	-1
4 hrs	+100	+28	+2	-7	0	0
24 hrs	+236	+91	+2	-8	0	0
3 days	+406	+186	+12	+5	+1	0
7 days	+607	+250	+26	+8	+2	+1
14 days	+772	+374	+38	+14	+2	0

NOTES: 1. "Initial" column indicates condition as removed from media.
2. "One Week" data indicates condition after 1 week air dry.

DIMENSIONAL CHANGES RESULTING FROM SOLVENT EXPOSURE

Material HYSTL	Δ Wt		Δ Length		Δ Thickness	
	Init	1 Week	Init	1 Week	Init	1 Week
	x 10^{-4} gr	x 10^{-4} gr	x 10^{-3} in.	x 10^{-3} in.	x 10^{-3} in.	x 10^{-3} in.
Distilled Water						
1 hr	+4	+2	0	-10	0	-2
4 hrs	0	-2	-1	0	+1	-1
24 hrs	+2	-1	-1	-12	0	+3
3 days	+2	+1	0	-3	-1	-6
7 days	-	-	+5	-2	-5	-5
14 days	-	-	-22	-12	+2	+2
Freon TF						
1 hr	-3	0	-1	+1	0	0
4 hrs	-1	0	0	+3	0	0
24 hrs	+3	+2	0	+1	0	-2
3 days	+3	+5	0	+5	0	+6
7 days	-	-	+2	+2	+6	+6
14 days		-	+8	+8	-1	+1
Isopropyl Alcohol						
1 hr	+3	+4	0	+5	0	+3
4 hrs	+1	+1	+1	-16	0	+2
24 hrs	+5	+2	0	+9	0	-1
3 days	+6	+1	0	-4	-1	0
7 days	-105	-107	-6	-8	0	-2
14 days	-45	-48	+12	+12	-6	-6
Trichlorethylene						
1 hr	+304	+107	+1	+2	-3	0
4 hrs	+510	+188	+4	0	+3	0
24 hrs	+1347	+571	+19	+3	+8	0
3 days	*	*	*	*	*	*
7 days	*	*	*	*	*	*
14 days	*	*	*	*	*	*

*Samples deteriorated

NOTES: 1. "Initial" column indicates condition as removed from media.
2. "One Week" data indicates condition after 1 week air dry.

DIMENSIONAL CHANGE RESULTING FROM SOLVENT EXPOSURE

Material EPT/Hydrin 263-2	Δ Wt		Δ Length		Δ Thickness	
	Init	1 Week	Init	1 Week	Init	1 Week
	x 10^{-4} gr	x 10^{-4} gr	x 10^{-3} in.	x 10^{-3} in.	x 10^{-3} in.	x 10^{-3} in.
Distilled Water						
1 hr	+322	-34	+7	-9	0	0
4 hrs	+758	-87	+18	-9	0	-2
24 hrs	+1691	-226	+100	-13	+4	-6
3 days	+1818	-269	+92	-21	+4	-5
7 days	+1886	-377	+92	-16	+4	-6
14 days	+1822	-413	+96	-29	+4	-6
Freon TF						
1 hr	+84	+4	-3	0	0	0
4 hrs	+209	-2	-24	-29	-1	-2
24 hrs	+422	+33	-4	-11	+2	+1
3 days	+685	+59	+17	-6	+1	0
7 days	+986	+23	+14	+5	+3	+1
14 days	+1162	+137	0	-19	+3	-1
Isopropyl Alcohol						
1 hr	+116	-14	-15	-20	0	-1
4 hrs	+206	+3	-11	-14	+1	-1
24 hrs	+494	-43	+31	+3	+3	+1
3 days	+753	-74	+58	-20	+2	-1
7 days	+772	-204	+50	-17	+2	-1
14 days	+774	-239	+75	-21	+4	0
Trichlorethylene						
1 hr	+1039	-26	+298	-14	+21	-1
4 hrs	+24488	-114	+677	-12	+39	-2
24 hrs	+33402	-352	+871	-29	+54	-3
3 days	+35193	-443	+793	-25	+62	-3
7 days	+36145	-576	+860	-45	+59	-3
14 days		-617	+886	-35	+56	-4

NOTES: 1. "Initial" column indicates condition as removed from medi
2. "One Week" data indicates condition after 1 week air dry.

DIMENSIONAL CHANGE RESULTING FROM SOLVENT EXPOSURE

Material 316-1 Fluorosilicone	Δ Wt Init x 10^{-4} gr	Δ Wt 1 Week x 10^{-4} gr	Δ Length Init x 10^{-3} in.	Δ Length 1 Week x 10^{-3} in.	Δ Thickness Init x 10^{-3} in.	Δ Thickness 1 Week x 10^{-3} in.
Distilled Water						
1 hr	+14	+6	-2	+6	0	-1
4 hr	+29	+7	-3	-5	-1	-1
24 hrs	+64	-4	-3	-5	0	0
3 days	+81	+6	+14	-4	+1	0
7 days	+116	+4	+9	+2	-1	-2
14 days	+122	+5	+7	+3	0	-1
Freon TF						
1 hr	+412	-44	+63	+1	+3	0
4 hrs	+2490	-85	+95	-14	+5	0
24 hrs	+2605	-194	+110	-20	+7	-1
3 days	+2509	-203	+107	-6	+6	-1
7 days	+2559	-204	+113	-9	+6	0
14 days	+2578	-200	+110	-14	+7	+1
Isopropyl Alcohol						
1 hr	+48	-18	+2	-6	-2	-3
4 hrs	+107	-22	+11	-3	+1	+1
24 hrs	+157	-82	+4	0	+1	0
3 days	+127	-87	+18	-2	0	0
7 days	+82	-50	+12	-5	+1	0
14 days	+28	-192	+4	-9	+1	0
Trichlorethylene						
1 hr	+1131	-43	+53	-2	+3	0
4 hrs	+1592	-101	+85	-2	+3	-2
24 hrs	+1504	-168	+65	-18	+4	-1
3 days	+1485	-223	+75	-16	+4	0
7 days	+1468	-231	+82	+4	+4	0
14 days	+1478	-240	+69	-13	+5	+1

NOTES: 1. "Initial" column indicates condition as removed from media.
2. "One Week" data indicates condition after 1 week air dry.

DIMENSIONAL CHANGES RESULTING FROM SOLVENT EXPOSURE

Material AF-E-71-2	Δ Wt		Δ Length		Δ Thickness	
	Init	1 Week	Init	1 Week	Init	1 Week
	10^{-4} gr	10^{-4} gr.	10^{-3} in.	10^{-3} in.	10^{-3} in.	10^{-3} in.
Distilled Water						
1 hr	+4	-7	-5	-10	+1	+1
4 hrs	+6	+15	-3	-10	0	0
24 hrs	+18	+3	-3	-7	0	0
3 days	+23	+15	-6	-11	+1	+1
7 days	+32	-4	-5	- 5	0	0
14 days	+35	+1	-7	-12	+1	+1
Freon TF						
1 hr	+1245	+34	+45	+2	+3	0
4 hrs	+3084	-8	+93	-8	+6	0
24 hrs	+5769	-113	+201	-19	+16	0
3 days	+5463	-233	+210	-23	+14	-1
7 days	+5536	-260	+189	-23	+14	-2
14 days	+5534	-238	+213	-33	+14	-2
Isopropyl Alcohol						
1 hr	+7	-3	-2	-6	0	-1
4 hrs	+24	-7	-10	-6	0	+1
24 hrs	+60	-14	+7	0	0	-1
3 days	+91	-40	+6	-9	0	0
7 days	+123	-70	+11	-8	0	0
14 days	+96	-103	+9	-8	0	-1
Trichlorethylene						
1 hr	+792	-105	+352	-12	+20	-1
4 hrs	+1341	-165	+452	-23	+25	0
24 hrs	+1859	-260	+456	-35	+27	-2
3 days	+1276	-269	+447	-33	+26	-2
7 days	+843	-268	+422	-23	+28	-1
14 days	+2047	-281	+455	-30	+27	-2

NOTES: 1. "Initial" column indicates condition as removed from media.
2. "One Week" data indicates condition after 1 week air dry.

Dimensional Change
After 7-Day Exposure

Material
AF-E-124D

	Weight Change		Length Change		Thickness Change	
	Init.	One Week	Init.	One Week	Init.	One Week
	grams					
Distilled Water	0.1603	0.1601	0.3230	0.324	0.0540	0.055
Freon TF	0.2442	0.1551	0.4103	-	0.0601	0.058
IPA	0.1553	0.1891	0.3403	0.342	0.0528	0.054
Trichlorethylene	0.1764	0.1739	0.3358	0.334	0.0549	0.055

Mechanical Properties Summary from Solvent and Temperature Exposure Test

Material	16 Hours		3 Days		10 Days		21 Days		1 Day		3 Days		7 Days		14 Days		Misc	
	Elongation (Percent)	Ultimate (psi)	Elongation (Percent)	Ultimate (psi)	Elongation (Percent)	Ultimate (psi)	Elongation (Percent)	Ultimate (psi)	Elongation (Percent)	Ultimate (psi)	Elongation (Percent)	Ultimate (psi)	Elongation (Percent)	Ultimate (psi)	Elongation (Percent)	Ultimate (psi)	Elongation (Percent)	Ultimate (psi)
AF-E-124																		
250°F					225	1,812												
325°F					205	1,730												
400°F					230	2,000												
Freon TF																		
Dist. Water									(Insufficient quantity of materials to conduct tests)									
IPA																		
Trichlorethylene																		
Teflon TFE																		
250°F	215	2,800	215	2,800	245	2,955	235	2,915										
325°F	210	2,950	185	2,600	220	3,080	187	2,735										
400°F	222	3,006	215	2,700	215	2,800	215	2,950										
Freon TF									193	2,711	211	2,933	232	3,067	255	2,864		
Dist. Water									255	2,933	225	3,067	185	2,711	240	2,800		
IPA									200+	2,889	200+	3,111	200+	3,156	200+	3,133		
Trichlorethylene									180	2,711	200	2,889	200	2,889	205	2,889		

AF-E-71-7

250°F	102	2,350		1,915	29	1,015	24	1,067										
325°F	31	895		760	11	470	10	297										
400°F	15	490		182	8	100	6	154										
Freon TF									120	2,516	11(							
Dist. Water									55	709	118	2,277	110	1,977	90	1,541	*150	709
IPA									125	2,361	124	2,431	98	1,694	105	1,904	*173	1,431
Trichlorethylene									120	2,346	75	1,435	112	2,050	120	2,333	*135	1,049

310-1 Phenyl Silicone

250°F	135	684	152	763	131	741	140	767
325°F	136	838	125	810	120	810	95	770
400°F	150	803	103	795	70	700	47	660
Freon TF								
Dist. Water								
IPA								
Trichlorethylene								

									160	870	138	793	132	808	123	707	*143	889
									135	790	143	840	153	840	155	807	*190	560
									140	840	128	774	155	897	130	790		
									108	660	140	902	150	911	124	741		

316-1 Fluorosilicone

250°F	167	700	165	695	145	640	155	615
325°F	158	720	175	805	130	622	118	570
400°F	152	728	115	585	93	512	80	419
Freon TF								
Dist. Water								
IPA								
Trichlorethylene								

									178	842	130	632	175	827	148	662	*195	910
									160	716	208	921	193	914	195	921	*165	841
									198	932	170	789	188	925	185	902		
									118	652	120	726	115	741	165	1,008	*155	741

VITON A

250°F	365	1,925	390	2,175	325	2,050	335	2,140
325°F	335	2,200	335	2,150	307	2,270	270	2,300
400°F	325	2,300	300	2,470	300	2,400	300	2,495
Freon TF								
Dist. Water								
IPA								
Trichlorethylene								

									350	1,669	388	1,639	370	1,504	368	1,474	*335	1,880
																	**348	1,820
									395	1,925	350	1,805	285	1,729	273	1,759	*357	2,106
																	**360	1,977
									400	1,798	285	1,433	395	1,645	400	1,579	*367	1,944
																	**370	1,812
									348	1,708	390	1,679	395	1,591	420	1,576	*370	1,870
																	**388	1,811

EPT-HYDRIN ***

250°F	690	2,690	605	2,203	485	2,632	370	1,970
				2,816		2,203		
325°F	260	140	-	-	-	-	-	-
400 F	-	-	-	-	-	-	-	-
Freon TF								
Dist. Water								
IPA								
Trichlorethylene								

									720	2,385	725	2,412	700	2,331	716	2,385	*708	2,136
																	**683	2,137
									690	2,111	703	2,100	717	2,278	750	2,222	*706	2,084
																	**705	1,853
									695	2,310	710	2,387	725	2,387	708	2,428	*722	2,320
																	**748	2,690
									735	2,189	750	2,469	773	2,329	735	2,469	*760	2,708
																	**725	2,100

HYSTL	F	Mod. of Rupture	E	Mod. of Rupture	E	Mod. of Rupture	F	Mod of Rupture	F	Mod. of Rupture	E	Mod. of Rupture	F	Mod. of Rupture	F	Mod. of Rupture		
250°F	324,684	9,394	333,608	9,682	302,686	8,818	352,861	1,861										
325°F	321,246	8,458	369,762	8,278	357,720	7,090	307,123	4,107										
400°F	331,701	6,856	322,893	6,476	312,211	6,478	336,841	6,227										
Freon TF									1,020,052	6,244	571,240	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		
Dist. Water									1,005,752	5,111	885,347	6,143	[illegible]	[illegible]	1,244,154	4,148		
IPA										[illegible]		[illegible]		[illegible]				
Trichlorethylene									1,182,[illegible]	[illegible]	[illegible]	[illegible]			[illegible]	4,244		

*1 Hour
**4 Hours
***Specimens physically decomposed at elevated temperatures